



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Alexander P. Moravsky, et al.

Examiner: Ashok Patel

Serial No: 09/680,291

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Docket: 21088/14311

For: DOUBLE-WALLED CARBON
NANOTUBES AND METHODS FOR
PRODUCTION AND APPLICATION

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Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

DECLARATION OF ALEXANDER P. MORAVSKY

I, Alexander P. Moravsky, hereby declare and state as follows:

1. I am one of the applicants of the above-identified application, and I have complete knowledge of all aspects of the application.
2. I am currently employed as Senior Scientist at MER Corporation, Tucson, Arizona, and have been so employed since 1999. Previous to that year, I assisted MER Corporation in Tucson, Arizona for several months in 1998 as a consultant. My employment history since 1975 is set forth in the Resume of Alexander P. Moravsky, attached hereto as Exhibit 1 and incorporated herein by reference and made a part hereof.
3. I am currently involved in fullerene and carbon nanotube materials synthesis and property studies, and have been so involved since 1991. I have extensive experience and many publications in this field, as set forth in my attached Resume in Exhibit 1.

4. I have earned Ph.D, M.Sc. and B.Sc. degrees in the fields of Chemistry, Physics and Molecular and Chemical Physics, all as set forth in my attached Resume in Exhibit 1.

5. The present application is directed to, *inter alia*, a solid substance comprised by more than one half by weight of hollow carbon nanotubes having walls consisting essentially of two layers of carbon atoms, said nanotubes consisting of two concentric nearly cylindrical graphene layers.

6. I have been advised by counsel that the United States Patent and Trademark Office ("USPTO") has considered the following claims as patentably distinct from the subject matter described in the previous paragraph:

108. Substantially pure double-wall nanotubes, whereas the double wall nanotubes have two concentric nearly cylindrical graphene layers.

109. The double-wall nanotubes according to Claim 108 which additionally contain single-wall nanotubes, wherein the ratio of double wall nanotubes to single wall nanotubes is greater than 30.

110. The double wall nanotube according to Claim 108 wherein the outer diameter of the double-wall nanotubes ranges from 3.4 to 5.5 nm, and the inner diameter ranges from 2.7 to 4.7 nm.

7. The subject matter described in Paragraph 5 encompasses the subject matter in Paragraph 6. More specifically, the subject matter in Paragraph 5 is directed to a solid which is comprised of more than 50% by weight of double wall nanotubes, where the nanotubes consist of two concentric nearly cylindrical graphene layers, while Claims 108-110 comprise substantially pure double wall nanotubes, where the double wall nanotubes have two concentric nearly

cylindrical layers. Thus, a product, which is substantially pure double wall nanotubes, as recited in paragraph 6, is encompassed by the subject matter in Paragraph 5.

8. I have also been advised by counsel that the USPTO in the latest Office Action has cited U.S. Patent No. 6,743,408 to Lieber et al. ("Lieber et al."). I have been advised by counsel that Lieber et al. filed a provisional application on September 29, 2000 to which it is claiming priority. For purposes of this Declaration, it is assumed that September 29, 2000 is the effective filing date of Lieber et al.

9. The present application was filed in the USPTO on October 6, 2000, which date is approximately 7 days after the earliest filing of Lieber et al.

10. The invention described and claimed in the above-identified application was completed in the U.S. prior to September 29, 2000.

11. As evidence hereof, annexed hereto and made a part hereof are Exhibits 1-11. All of the acts described herein took place in either the United States or a NAFTA country other than the United States or a WTO member country prior to September 29, 2000, and were performed by myself or by scientists and/or technicians working under my direct supervision or control, except to the extent indicated herein. Data or information not pertinent to the invention and dates have been masked out in the preparation of their photocopies.

12. Attached hereto as Exhibit 2 is a copy of a notebook page, which outlines a synthesis for a product of the present invention. This outlines an arc discharge evaporation of a metal-graphite electrode, where the anode was a graphite rod (8.2 mm in diameter), having a drilled channel of about 189 mm in length and 3.16 mm in diameter. The cathode was a graphite rod, which was positioned coaxially with the anode. The catalyst was prepared by heating nickel, cobalt and iron together with elemental sulfur. The resulting conglomerate was ground in

a ball mill to a micron particle size and then intimately mixed with carbon powder. The channel drilled in the graphite rod was tightly filled with the mixture to obtain an anode containing 79.91% C, 10.26% Ni, 2.77% Co, 5.44% Fe and 1.62%S.

An arc discharge was carried out with an arc current of 81A at 350 Torr in an atmosphere of Argon (175 Torr) and hydrogen (175 Torr) (1:1). The voltage drop across the gap was 26-28 V, and the rod feed was 2 mm/min. The duration of the arc process was about 45 min.

The carbon material produced in the arc discharge was deposited on the walls of the reactor and on the electrodes wherefrom it was collected separately. A fibrous material (elastic web-like product) was collected from the cathode.

13. A SEM examination of the web-like products obtained from the process described in Paragraph 12 showed dominating amounts of curled fibers being present in the product.

14. Exhibit 3 is an example of a TEM image of the product obtained from the process described in Paragraph 12. As shown in Exhibit 3, unusually thick nanotubes of approximately 4 nm in diameter were observed, assembled in thin bundles containing a few tubes.

15. The image was quite different than the appearance of single walled nanotubes, commonly produced in arc discharge processes, whereby tubes are about 1.5 nm in diameter and the bundles contain some dozen tubes.

16. Further, the walls of the tubes in Fig. 3 are thicker than is observed for commonly produced SWNTs, which is consistent with the nanotubes having two walls.

17. TEM images made by an instrument with better resolution unambiguously show that the tubes produced by the process described hereinabove in paragraph 12 have a two-wall structure. The images are attached hereto as Exhibits 4-8. These images unambiguously show that the nanotubes have two concentric nearly cylindrical graphene layers.

18. Additional samples were prepared in accordance with the procedure outlined in Example 1.

19. Samples, which were prepared by me or under my direct supervision, in accordance with the procedure described in the underlying application, such as Example 1, were examined under HRTEM (High Resolution TEM) in Russia by my colleague, Dr. Krinichnaja. Attached as Exhibit 9 is an email message from Dr. Krinichnaja in Russian; Exhibit 10 is a translation thereof, which verifies that the sample which I provided to her and which she examined using TEM, consisted of double wall nanotubes, having diameters of 3.5 to 5.2 nm. Good quality HRTEM analysis of the samples produced prior to September 29, 2000 revealed that samples contained not only DWNTs as the dominant carbon product, and also that no SWNTs were detected.

20. As further evidence, I enclose a copy of an article authored by, *inter alia*, Dr. Loutfy and myself that was published in Carbon 39, 2001, 761-770, which is attached as Exhibit 11. Although the article was published in 2001, it was received by the publisher on June 2, 2000, and accepted for publication on July 24, 2000, both dates of which are prior to the September 29, 2000 date of the earliest filing of Lieber et al. All of the experiments and the data described in the paper referred to in Exhibit 11 were conducted prior to the September 29, 2000 date of the earliest filing of Lieber et al.

21. Although the paper in Exhibit 11 mainly refers to a HRTEM study of DWNTs, it also provides a description of the preparation of DWNTs. The preparation of the nanotubes was conducted by Dr. Loutfy and/or myself or under our direct supervision, and the synthesis is in accordance with the procedure we described in our underlying application. As shown by the

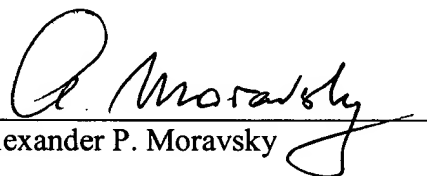
HRTEM micrographs, the synthesis produced double wall nanotubes having two concentric nearly cylindrical graphene layers.

22. As shown by all the data described herein, all of the experiments described herein produced a solid product comprised by more than one half by weight of hollow carbon nanotubes having walls consisting essentially of two layers of carbon atoms, consisting of two concentric nearly cylindrical graphene layers. All of the experiments described herein were performed prior to September 29, 2000.

23. Thus, the sample of solid product described in the underlying application which is comprised of more than one half by weight of hollow carbon nanotubes having walls consisting of two layers of carbon atom, and consisting of two concentric nearly cylindrical graphene layers were synthesized and analyzed prior to the September 29, 2000, the earliest filing date of Lieber et al.

24. I further declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: October 25, 2007


Alexander P. Moravsky

RESUME
ALEXANDER P. MORAVSKY

EDUCATION

Ph.D. Chemistry, Moscow Institute for Physics and Technology (MIPT), 1975
M.Sc. Physics of Fast Processes, MIPT, 1972
B.Sc. Molecular and Chemical Physics, MIPT, 1970

PROFESSIONAL EXPERIENCE

1998-Present MER Corporation, Senior Scientist, Tucson, AZ
1980-1998 Senior Research Fellow, Head of a Research Group, Institute of Problems
of Chemical Physics, Chernogolovka, Russia
1979-1980 Post Doctorate of Prof. J.K.Stille, Colorado State Univ., Fort Collins, CO
1975-1979 Research Fellow, Institute of Chemical Physics, Chernogolovka, Russia

RECORD OF TECHNICAL ACHIEVEMENTS

- 11 Patents
- 230 papers in refereed journals
- 5 Reviews

INDUSTRIALLY IMPLEMENTED TECHNOLOGIES:

- technique for ketones and aldehydes micro-analysis
- methane absorption accumulators

AREAS OF TECHNICAL EXPERTISE

- R&D on advanced new materials, basic chemical technology, organic synthesis
- Catalysts, small molecule catalytic conversions of industrial interest
- Redox processes and fast reactions in solution and films
- Purification of substances, analytical chemistry
- Analysis of sophisticated chemical kinetic schemes
- Spectral analysis, NMR and ESR, XPS, GC, GC/MS, LC, HPLC
- Fullerene and nanocarbon materials science and technology

Dr. Moravsky A.P.
List of recent publications. 1993-2007.

1. "The Loss-Function of Solid C₆₀", Synthetic Metals, v.56, No 2-3, 2961-2966 (1993). V.I.Rubtsov, Yu.M.Shulga, A.S.Lobach, and A.P.Moravsky.
- "Photophysical Properties of C₆₀. Picosecond Study of Intersystem Crossing", J.Photochem.Photobiol. A: Chem., v.70, No2, 153-156 (1993). V.A.Nadtochenko, I.V.Vasil'ev, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, A.P.Moravskii, and A.F.Shestakov.
- "Photophysical properties of C₇₀. Picosecond Laser Photolysis". Zh. Fiz.Khimii, N9, 1880-1883 (1993). I.V.Vasiliev, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, V.A.Nadtochenko, and A.P.Moravskii.
- "Photochemical Properties of C₆₀. Triplet Excited C₆₀ Quenching by Electron Acceptors TCNQ and TCNE in Solution. Laser Photolysis Study", Chem.Phys.Letters, v.208, No5-6, 431-435 (1993), V.A.Nadtochenko, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, and A.P.Moravskii.
- "Triplet Excited C₆₀ Quenching by TCNE in Benzonitrile Solution. Formation of the Ion-Radical Pair [C₆₀⁺...TCNE]", Russian Chemical Bulletin, v.42, N7, 1171-1173 (1993). V.A.Nadtochenko, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, and A.P.Moravskii.
- "Photophysical Properties of Fullerenes. Picosecond and Nanosecond Transient Absorption Spectra of C₆₀ and C₇₀. New Bands in the Near IR." Fullerenes. The First Int. Interdisciplinary Colloquium on the Sci. and Tech. of Fullerenes, 1993, Santa Barbara, USA, Abstract, p.116. Nadtochenko V.A. et al.
- "Photochemical Properties of C₆₀. Photooxidation of C₆₀ by TCNE and TCNQ in Solutions. Laser Photolysis Study". Fullerenes. The First Int. Interdisciplinary Colloquium on the Sci. and Tech. of Fullerenes, 1993, Santa Barbara, USA, Abstract, p.192-194. Nadtochenko V.A. et al.
- "Photophysical and Photochemical Properties of C₆₀. Picosecond and Nanosecond Laser Photolysis Study", International Workshop on Fullerenes and Atomic Clusters", 1993, St.Peterburg, Russia, p.29. Nadtochenko V.A. et al.
- "On the Reaction of Buckminsterfullerene with Tetrabutylammonium Tetrahydroborate", Russian Chemical Bulletin, N3, 803(1993). V.D.Makhaev, Yu.M.Shulga, A.S.Lobach, V.N.Vasilets, O.S.Roshchupkina, and A.P.Moravsky.
10. "Reaction of Organic Cation Tetrahydroboronates with Fullerene C₆₀", Int. Conference "Physics and Chemistry of Fullerenes", Greece, 1993, Abstracts, p.34. V.D.Makhaev, A.S.Lobach, Yu.M.Shulga, O.S.Roshchupkina, and A.P.Moravsky.
- "Interaction of Methane with Acetylene in the Presence of Ziegler-Natta Type Catalysts", Neftekhimiya, v.32, N4, 324-330 (1992). E.M.Efimova, A.P.Moravskii, and N.F.Noskova.
- "Characteristic Loss Spectrum, Accompanying the C_{1s} Photoelectron Peak of Fullerene C₆₀", JETP Letters, v.55, No.2, 132-135 (1992), Yu.M.Shulga, A.P.Moravsky, A.S.Lobach, and V.I.Rubtsov.
- "Study of the Single-Fold Electron Energy Losses Spectra of Individual Fullerenes C₆₀ and C₇₀ and of "Graphite-like materials", Doklady Akad.Nauk., v.325, N4, 779-781 (1992). Yu.M.Shulga, A.P.Moravsky, A.S.Lobach, and V.I.Rubtsov.
- "First-Order Phase Transition with a Large Change in Volume in the Fullerene C₆₀ Under Pressure", JETP Lett, v.59, N4, 279-282 (1994). I.O.Bashkin, V.I.Rashchupkin, N.P.Kobelev, A.P.Moravskii, Ya.M.Soifer, and E.G.Ponyatovskii.
- "Reactions of Hydrocarbons with Electrophilic Transition Metal Complexes in Trifluoroacetic Acid Media", Uspekhi Khimii, v.63, N2, 130-144 (1994). N.F.Goldshleger, and A.P.Moravskii.
- "Orientational Rotation of C₆₀ Molecules in Different Solutions", JETP Letters, v.60, No5, 320-325 (1994). I.V.Rubtsov, D.V.Khudyakov, V.A.Nadtochenko, A.S.Lobach, and A.P.Moravskii.
- "Quenching of Triplet-Excited Fullerene C₆₀ by TCQM in Solutions", Zh.Fiz.Khimii, v.68, N2, 228-231 (1994). V.A.Nadtochenko, N.N.Denisov, A.S.Lobach, and A.P.Moravskii.
- "Interaction of Fullerene C₆₀ with 3-Amino-1-Propanol", Russian Chemical Bulletin, N6, 1143 (1996). N.F.Goldshleger, A.S.Lobach, A.S.Astakhova, M.G.Kaplunov, A.V.Kulikov, A.P.Moravskii, O.S.Roshchupkina, and Yu.M.Shulga.
- "Synthesis of Organic Ferromagnetics by Pyrolysis of Conducting Polymers", Int.Conf.Synthetic Metals, Korea, 488 (1994), B.Z.Lubentsov, A.P.Moravsky, T.M.Moravskaya, and G.I.Zvereva.
20. "A New Phase Transition in the T-P Diagram of C₆₀ Fullerite", J.Phys.: Condens. Matter, v.6, 7491-7498 (1994), I.O.Bashkin, V.I.Rashchupkin, A.F.Gurov, A.P.Moravsky, O.G.Rybchenko, N.P.Kobelev, Ya.M.Soifer, and E.G.Ponyatovskiy.
- "Acoustic Properties of Fullerene Compacts", Molecular Materials, v.4, 139-141 (1994), I.O.Bashkin, N.P.Kobelev, Ya.M.Soifer, and A.P.Moravsky.
- "Dissipative Properties of Fullerene-C₆₀", Int.Workshop Fullerenes and Atomic Clusters, 54 (1994), I.O.Bashkin, N.P.Kobelev, A.P.Moravsky, and Ya.M.Soifer.
- "Photophysical and Photochemical Properties of C₆₀ and C₇₀. Intersystem Crossing in Solutions. Photooxidation of T₁-excited C₆₀ by Electron Acceptors in Solutions", Molecular Materials, v.4, No1-3, 95-103 (1994), V.A. Nadtochenko, N.N. Denisov, I.V.Vasiliev, I.V.Rubtsov, A.S.Lobach, and A.P.Moravsky.
- "The Picosecond and Nanosecond Laser Photolysis Techniques Study of Photophysical and Photochemical Properties of Fullerenes", Int.Workshop Fullerenes and Atomic Clusters, 54 (1994), V.A.Nadtochenko, I.V.Vasiliev, I.V.Rubtsov, A.S.Lobach, and A.P.Moravsky.

- "Rotational Reorientation Dynamics of C_{60} in Various Solvents. Picosecond Transient Grating Experiments", In "Fullerenes. Recent Advances in the Chemistry and Physics of Fullerenes and Related Materials", eds. K.M.Kadish, R.S.Ruoff. The Electrochemical Society, Pennington, New Jersey, 1994, p.1619-1627, I.V.Rubtsov, D.V.Khudiakov, V.A.Nadtochenko, A.S.Lobach, and A.P.Moravsky.
- "External Heavy Atom Effect on the Intersystem Crossing of Fullerenes. Picosecond Laser Photolysis Study". In " Fullerenes. Recent Advances in the Chemistry and Physics of Fullerenes and Related Materials", eds. K.M.Kadish, R.S.Ruoff. The Electrochemical Society, Pennington, New Jersey, 1994, p.1658-1669, V.A.Nadtochenko, I.V.Vasilev, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, and A.P.Moravsky.
- "Rotational Reorientation Dynamics of C_{60} in Various Solvents. Picosecond Transient Grating Experiments", Chem.Phys.Lett., v.229, Nos 4 and 5, 517 (1994), I.V.Rubtsov, D.V.Khudiakov, V.A.Nadtochenko, A.S.Lobach, and A.P.Moravsky.
- "Reduction of Fullerene C_{60} with 3-Amino-1-Propanol", Molecular Materials, v.4, N1-3, 185-186 (1994), N.F.Goldshleger, A.S.Lobach, M.G.Kaplunov, A.V.Kulikov, A.P.Moravsky, O.S.Roshchupkina, and Yu.M.Shulga.
- "Investigation of Fullerenes C_{60} , C_{70} Reduction in Neat Primary Amines", MRS Fall Meeting, Abstracts, Boston, USA, G5.42 (1994). A.S.Lobach, N.F.Goldshleger, M.G.Kaplunov, A.V.Kulikov, and A.P.Moravsky.
30. "Interaction of Fullerene C_{60} with 3-Amino-1-Propanol", IW Fullerenes and Atomic Clusters, 73 (1994), N.F.Goldshleger, A.S.Lobach, M.G.Kaplunov, A.P.Moravsky, O.S.Roshchupkina, Yu.M.Shulga, and A.V.Kulikov.
- "Elastic and Dissipative Properties of Fullerite", Solid State Physics, v.36, N9, 2732-2737 (1994). N.P.Kobelev, A.P.Moravsky, Ya.M.Soifer, I.O.Bashkin, and O.G.Rybchenko.
- "Internal Friction and Sound Wave Velocities in Pure C_{60} Fullerite", Physica Status Solidi, B - Basic Research, 190:1, 157-162 (1995) N.P.Kobelev, Ya.M.Soifer, I.O.Bashkin, A.F.Gurov, A.P.Moravsky, and O.G.Rybchenko.
- "Method of Determination of Fe-Protein Concentration in the Solution and its Application in the Study of the Kinetics of Nitrogenase Reaction". Biokhimiya, v.60, 1512-1520. S.Yu.Druzhinin, L.A.Syrsova, A.V.Khramov, A.P.Moravskii, and N.I.Shkondina.
- "Fullerene C_{60} Orientational Dynamics Study in Different Solvents by Picosecond Non-Stationary Grating Technique". Khimicheskaya Fizika, v.14, N14, 96-104 (1995). I.V.Rubtsov, D.V.Khudyakov, V.A.Nadtochenko, A.S.Lobach, and A.P.Moravskii.
- "UV-vis Photometric Analysis of Fullerenes C_{60} and C_{70} in Toluene and Hexane Solutions", in: Fullerenes. Recent Advances in the Chemistry and Physics of Fullerenes and Related Materials, v.2, Ed. K.M.Kadish and R.S.Ruoff, 1995, pp.156-169. A.P.Moravsky, P.V.Fursikov, L.M.Kachapina, A.V.Khramov, and N.V.Kiryakov.
- "A Nearly Orthorhombic Phase of C_{60} . Properties and the Stability Region in the T-P Diagram", in: Fullerenes. Recent Advances in the Chemistry and Physics of Fullerenes and Related Materials, v.2, Ed. K.M.Kadish and R.S.Ruoff, 1995, pp.952-963. A.P.Moravsky, G.E.Abrosimova, I.O.Bashkin, R.A.Dilanian, A.F.Gurov, N.P.Kobelev, V.I.Rashchupkin, O.G.Rybchenko, Ya.M.Soifer, V.Sh.Shekhtman, and E.G.Ponyatovsky.
- "A Study of Rotational Mobility of C_{60} Molecules in Different Solutions by the Induced Grating Technique", Chem.Phys.Reports, 1995, v.14, N 4, 490-502. I.V.Rubtsov, D.V.Khudyakov, V.A.Nadtochenko, A.S.Lobach, and A.P.Moravskii.
- "Vibrational Spectroscopic Study of a New Polymeric C_{60} Phase", Materials Research Society 1995 Fall Meeting, Abstracts, 1995, FF10.4, p.170, A.M.Rao, P.C.Eklund, I.O.Bashkin, E.G.Ponyatovsky, and A.P.Moravsky.
- "Photoluminescence Studies of a New Polymeric C_{60} Phase", Materials Research Society 1995 Fall Meeting, Abstracts, 1995, FF12.2, p.171, U.D.Venkateswaran, A.M.Rao, P.C.Eklund, I.O.Bashkin, E.G.Ponyatovsky, and A.P.Moravsky.
40. "Method for Assay of Fe-Protein Concentration and its Application in the Study of Nitrogenase Reaction Kinetics", Biochemistry, v.60, N9, pp.1153-1158 (1995), S.Yu.Druzhinin, L.A.Syrsova, A.V.Khramov, A.P.Moravsky, and N.A.Shkondina.
- "Photostimulation of Nitrogenase Reaction in Vitro for Investigation of Nitrogenase Mechanism Action", in: "Nitrogen Fixation: Fundamentals and Applications". Proceedings of the 10-th Int. Congress on Nitrogen Fixation, St.Peterburg, eds. Tikhonovich I.A., Provorov N.A., Romanov V.I., Newton W.E., Kluwer Academic Publishers, the Netherlands, 1995, pp.97-102, S.Yu.Druzhinin, L.A.Syrsova, A.V.Khramov, and A.P.Moravsky.
- "Internal Friction and Elastic Properties of a New Pressure-Induced Phase of C_{60} Fullerite", Int.Workshop Fullerenes and Atomic Clusters, 1995, p.29, N.P.Kobelev, Ya.M.Soifer, I.O.Bashkin, A.P.Moravsky, and E.G.Ponyatovsky.
- "On the Optimal Conditions for Fullerene Production in Direct Current Arc", Int. Workshop Fullerenes and Atomic Clusters, Abstracts, St.Peterburg, 1995, p.39-40. A.P.Moravsky, P.V.Fursikov.
- "Preparation and Single Crystal Structure Determination of the Intercalate C_{60} /TMPD", IW Fullerenes and Atomic Clusters, 1995, p.53-54, V.A.Nadtochenko, A.P.Moravsky, V.V.Gritsenko, G.V.Shilov, and O.A.Dyachenko.
- "Properties of Materials Encapsulated into Carbon Nanotubes", Int.Workshop Fullerenes and Atomic Clusters, 1995, p.64-65, A.F.Gurov, A.S.Aronin, I.O.Bashkin, A.P.Moravsky, V.E.Muradyan, V.M.Teplinsky, and E.G.Ponyatovsky.
- "Picosecond Dynamics of Charge Recombination in EDA Complexes between C_{60} , C_{70} and ternary Amines", IW Fullerenes and Atomic Clusters, 1995, p.109-110, N.N.Denisov, I.V.Rubtsov, A.S.Lobach, A.P.Moravsky, and V.A.Nadtochenko.
- "UV-VIS Molar Absorption Coefficients for C_{60} and C_{70} in Toluene and Hexane", IW Fullerenes and Atomic Clusters, 1995, p.111-112, A.P.Moravsky, P.V.Fursikov, A.V.Khramov, and N.V.Kiryakov.
- "IR Spectroscopy of C_{60} /TMPD and C_{60} /TPA Single Crystals. Photoinduced Absorption in Crystals of C_{60} with tertiary Amines", IW Fullerenes and Atomic Clusters, 1995, p.120-121, A.V.Bazhenov, T.N.Fursova, A.P.Moravsky, and V.A.Nadtochenko.

- "A High-Pressure Study of Phase Transformations in C₆₀ Fullerite", IW Fullerenes and Atomic Clusters, 1995, p.125-126, I.O.Bashkin, A.F.Gurov, A.P.Moravsky, V.I.Rashchupkin, and E.G.Ponyatovsky.
50. "Investigation of Fullerenes C₆₀, C₇₀ Reduction in Neat Primary Amines", IW Fullerenes and Atomic Clusters, 1995, p.155-156, A.S.Lobach, N.F.Goldshleger, M.G.Kaplunov, A.V.Kulikov, and A.P.Moravsky.
- "Statistical Processing of the Spectra of Fullerene Black Extracts: The Constancy of the C₆₀/C₇₀ Ratio". Doklady Chemistry, v.351, Nos.1-3, pp.298-300 (1996). A.G.Ryabenko, A.A.Ryabenko, A.P.Moravskii, and P.V.Fursikov.
- "X-Ray Diffraction Study of a High Pressure Phase of Fullerite C₆₀". Poverkhnost, N7, 79-81 (1996). G.E.Abrosimova, I.O.Bashkin, R.A.Dilanian, A.P.Moravskii, E.G.Ponyatovskii, O.G.Rybchenko, and V.Sh.Shekhtman.
- "Reactions of Fullerenes with Deuterium in the Presence of Palladium", Russian Chemical Bulletin, v.45, No.7, 1778-79 (1996). B.P.Tarasov, V.N.Fokin, A.P.Moravsky, and Yu.M.Shulga.
- "Spectrum of Optical Phonons and Low-Energy Electron Transitions in Single Crystals of C₆₀/tetramethyl- p-phenylenediamine and C₆₀/triphenylamine", Russian Chemical Bulletin, v.45, No.6, 1388-1392 (1996). A.V.Bazhenov, M.Yu.Maksimuk, T.N.Fursova, A.P.Moravsky, and V.A.Nadtochenko.
- "Synthesis of a C₆₀/TMPD Complex and its Crystal Structure", Russian Chemical Bulletin, v.45, No.5, 1224-1225 (1996). V.A.Nadtochenko, V.V.Gritsenko, O.A.Dyachenko, G.V.Shilov, and A.P.Moravsky.
- "Molecular and Crystal Structure of a Charge-Transfer Complex C₆₀/TNPd" Zh.Fiz.Khimii, 1996. V.A.Nadtochenko, V.V.Gritsenko, O.A.Dyachenko, G.V.Shilov, and A.P.Moravskii.
- "Orientational Dynamics of C₇₀ Molecules in Chlorobenzene", Russian Chemical Bulletin, v.45, No.3, 560-563 (1996). D.V.Khudyakov, I.V.Rubtsov, V.A.Nadtochenko, and A.P.Moravsky.
- "Charge-Transfer Complexes of Fullerene C₇₀ and Ternary Amines in Chlorobenzene. Picosecond Dynamics of Charge Recombination", Russian Chemical Bulletin, v.45, No.5, 1091-1098 (1996). V.A.Nadtochenko, N.N.Denisov, I.V.Rubtsov, and A.P.Moravsky.
- "UV-vis Molar Absorption Coefficients for Fullerenes C₆₀ and C₇₀", Molecular Materials, v.7, 241-246 (1996). A.P.Moravsky, P.V.Fursikov, N.V.Kiryakov, and A.G.Ryabenko.
60. "High-Pressure Study of Phase Transformations in C₆₀ Fullerite", Molecular Materials, v.7, No.1-4, 271-276 (1996). I.O.Bashkin, A.F.Gurov, V.I.Rashchupkin, E.G.Ponyatovsky and A.P.Moravsky.
- "Internal Friction and Elastic Properties of a New Pressure-Induced Phase of C₆₀ Fullerite", Molecular Materials, v.7, No.1-4, 261-266 (1996). N.P.Kobelev, Ya.M.Soifer, I.O.Bashkin, A.P.Moravsky and E.G. Ponyatovsky.
- "Preparation and Single Crystal Structure Determination of the C₆₀/TMPD Electron Donor-Acceptor Complex", Molecular Materials, v.7, No.1-4, 103-104 (1996). V.A.Nadtochenko, A.P.Moravskii, V.V.Gritsenko, G.V.Shilov, and O.A.Dyachenko.
- "Nitrogenase Component Stoichiometry and the Possibility of Nitrogenase Dissociation as Studied by Stopped-Flow Spectrometry", Biochemistry (USA), 1996, S.Yu.Druzhinin, L.A.Syrtsova, A.V.Khranov, A.P.Moravsky, and N.A.Shkondina.
- "Raman and Infrared Studies of Pressure-Polymerized C₆₀", The 12th Yokohama 21st Century Forum, Ext.Abstr., 1996, p.52-61. A.M.Rao, P.C.Eklund, J-L.Hodeau, L.Marques, M.Nunez-Regueiro, J.Tucker, M.A.Duncan, I.O.Bashkin, E.G.Ponyatovsky, and A.P.Moravsky.
- "Synthesis of Fullerene and Alkaline Earth Hydrides at Mild Pressure-Temperature Conditions", Abstracts Book of Int.Symp. on Metal Hydrogen Systems, Switzerland, 1996, M1-19. B.P.Tarasov, V.N.Fokin, E.E.Fokina, S.P.Shilkin, A.P.Moravsky, Yu.M.Shulga, and V.A.Yartys.
- "Optimization of the Arc Synthesis of Fullerenes", 189th Meeting of The Electrochemical Society Inc., Ext.Abstr., Fullerenes S1, Los Angeles, 1996, p.112. A.Moravsky, P.Fursikov, A.Krestinin, and A.Ryabenko.
- "Magnetic Properties of Polyaniline", Int.Conf. Sci.Tech.Synth.Metals ICSM'96, Abstracts, Snowbird, Utah, 1996, p.37. B.Z.Lubentsov, A.P.Moravsky, G.I.Zvereva, O.V.Bochkova, V.N.Spector, and A.A.Ovchinnikov.
- "Production, Purification and Filling of Carbon Nanotubes", IX-th Trilateral Seminar on Superconductivity, Abstracts, Ilmenau, Germany, (1996), p.57-59. A.Gurov, A.Moravsky, R.Oganian, A.Aronin, and I.Bashkin.
- "Neutron Spectroscopy of C₆₀Hx Quenched under Hydrogen Pressure", Proc.First European Conf. on Neutron Scattering (ECNS'96), 1996, Interlaken, Switzerland, p.74-82. A.I.Kolesnikov, V.E.Antonov, I.O.Bashkin, E.G.Ponyatovsky, A.Yu.Muzychka, A.P.Moravsky, G.Grosse and F.E.Wagner.
70. "Neutron Scattering Study of a High-Pressure Polymeric C₆₀ Phase", J.Phys.: Condensed Matter, v.8, 10939-10949 (1996), A.I.Kolesnikov, I.O.Bashkin, A.P.Moravsky, M.A.Adams, M.Prager, and E.G.Ponyatovsky.
- "Orientational Behavior of C₇₀ Molecules in Chlorobenzene", Chem.Phys.Letters, v.249, Nos.1 and 2, 101-105 (1996), I.V.Rubtsov, D.V.Khudyakov, A.P.Moravsky, and V.A.Nadtochenko.
- "Elastic and Dissipative Properties of Solid C₆₀", J.de Physique, A58, 3431-3434 (1996). Ya.M.Soifer, N.P.Kobelev, I.O.Bashkin, A.P.Moravsky and E.G.Ponyatovsky.
- "Fullerene Hydrides: Synthesis, Properties and Structure", Russian Chemical Reviews, V.66, No.4, 323-342 (1997). N.F.Goldshleger, A.P.Moravsky.

- "Activation of Methane over Fullerene Black", *Neftekhimiya*, v.37, N2, pp.117-123 (1997). S.D.Kushch, A.P.Moravskii, V.E.Muradian, and P.V.Fursikov.
- "Electrochemical Polymerization of Fullerene C₆₀", *Russian Chemical Bulletin*, V.46, No.4, 830-831 (1997). A.P.Moravskii, I.O.Bashkin, O.N.Efimov, E.P.Krinichnaya, E.G.Ponyatovskii, and V.V.Strelets.
- "Conversions in the System Fullerene-Intermetallics-Hydrogen", *Zh.neorg.khimii*, v.42, N6, 920-922 (1997) B.P.Tarasov, V.N.Fokin, A.P.Moravskii, and Yu.M.Shulga.
- "Hydrogenation of Fullerenes in the Presence of Intermetallic Compounds or Metals", *Russian Chemical Bulletin*, v.46, No.4, 649-652 (1997). B.P.Tarasov, V.N.Fokin, A.P.Moravskii, and Yu.M.Shulga.
- "Magnetic Properties of Polyaniline", *Doklady Chemistry*, v.354, N5, 635-537 (1997). G.I.Zvereva, B.Z.Lubentsov, A.P.Moravskii, O.V.Bochkova, V.N.Spector, and A.A.Ovchinnikov.
- "Properties of C₆₀ Polymerized Under High Pressure and Temperature", *Applied Phys.A* 64, N4, 231-239 (1997). A.M.Rao, P.C.Eklund, U.D.Venkateswaran, J.Tucker, M.A.Duncan, G.Bendele, P.W.Stephens, J.-L.Hodeau, L.Marques, M.Nunez-Regueiro, I.O.Bashkin, E.G.Ponyatovsky, and A.P.Moravsky.
80. "Photoluminescence of Solid C₆₀ Polymerized Under High Pressure", *Chem.Phys.Lett.*, 272, 32-37 (1997). I.O.Bashkin, A.N.Izotov, A.P.Moravsky, V.D.Negrii, R.K.Nikolaev, Yu.A.Ossipyan, E.G.Ponyatovsky and E.A.Steinman.
- "Neutron Spectroscopy of C₆₀ Fullerite Hydrogenated under High Pressure; Evidence for Interstitial Molecular Hydrogen", *J. Phys.: Condensed Matter*, v.9, 2831-2838 (1997). A.I.Kolesnikov, V.E.Antonov, I.O.Bashkin, G.Grosse, A.P.Moravsky, A.Yu.Muzychka, E.G.Ponyatovsky and F.E.Wagner.
- "Neutron Spectroscopy of C₆₀H_x Quenched under Hydrogen Pressure", *Physica B: Condensed Matter*, v.234-236, 10-12 (1997). A.I.Kolesnikov, V.E.Antonov, I.O.Bashkin, E.G.Ponyatovsky, A.Yu.Muzychka, A.P.Moravsky, G.Grosse, and F.E.Wagner.
- "Hydrogenation of Fullerenes C₆₀ and C₇₀ in the Presence of Hydride Forming Metals and Intermetallic Compounds", *Journal of Alloys & Compounds*, v.253-254, 25-28 (1997). B.P.Tarasov, V.N.Fokin, A.P.Moravsky, Yu.M.Shulga, and V.A.Yartys.
- "A SEM Study of the Internal Structure of Cathode Deposits Containing Different Types of Carbon Nanotubes", 10th Russian Simp.SEM and Analyt.Techniques for Solid State Studies, Abstract, Chernogolovka, 1997, 82-83, A.P.Moravskii, A.B.Ormont.
- "Kinetics of Fullerene Formation in Arc Reactor", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.16. A.V.Krestinin, A.P.Moravsky.
- "Vibrational Spectra of C₆₀ Hydrofullerite Prepared Under High Hydrogen Pressure", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.22. I.O.Bashkin, A.I.Kolesnikov, V.E.Antonov, G.Grosse, A.P.Moravsky, A.Yu.Muzychka, F.E.Wagner, and E.G.Ponyatovsky.
- "Carbon Arc in Helium Produces Exactly 5 Molecules of C₆₀ per one of C₇₀", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.64. A.P.Moravsky, A.G.Ryabenko, A.A.Ryabenko, and P.V.Fursikov.
- "Vibrational Spectrum of a C₆₀ High-Pressure Polymer", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.75. I.O.Bashkin, A.I.Kolesnikov, M.A.Adams, A.P.Moravsky, M.Prager, and E.G.Ponyatovsky.
- "Photoluminescence of C₆₀ Crystals Polymerized under High Pressure", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.76. I.O.Bashkin, A.N.Izotov, A.P.Moravsky, V.D.Negrii, R.K.Nikolaev, Yu.A.Ossipyan, E.G.Ponyatovsky, and E.A.Steinman.
90. "Cathode Polymerization of Solid Films of Fullerene C₆₀", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.82. A.P.Moravsky, I.O.Bashkin, O.N.Efimov, E.P.Krinichnaya, E.G.Ponyatovsky, and V.V.Strelets.
- "Carbide Formation in the System Fullerene-Metal (Intermetallic Compound)-Hydrogen", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.128. B.P.Tarasov, V.N.Fokin, A.P.Moravsky, and Yu.M.Shulga.
- "Hydrogenation of Solid Compositions of Fullerenes with Metals or Intermetallics", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.152. B.P.Tarasov, V.N.Fokin, A.P.Moravsky, Yu.M.Shulga, and V.A.Yartys.
- "Curie Point of Nickel Encapsulated in Carbon Nanotubes", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.168. A.F.Gurov, A.S.Aronin, A.P.Moravsky, and A.D.Styrkas.
- "Electrochemical Behavior of Polymer Compositions Containing Fullerenes or Nanotubes", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.259. L.M.Zemtsov, G.P.Karpacheva, V.V.Kozlov, E.P.Krinichnaya, O.N.Efimov, A.P.Moravsky.
- "Fullerenes and Soot as Catalyst Supports in Propylene Polymerization", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.295. N.M.Galashina, A.N.Klyamkina, N.J.Kovalyova, A.P.Moravsky and G.A.Vinogradov.
- "Fullerene-Based Catalysts for Methane Dehydrogenation", 3rd Int.Workshop Fullerenes & Atomic Clusters, St.Petersburg, Russia, Book of Abstracts, 1997, p.298. S.D.Kushch, E.I.Knerelman, E.M.Koldasheva, V.L.Kuznetsov, V.E.Muradyan, P.V.Fursikov, A.V.Khranov, A.P.Khrushch, and A.P.Moravsky.
- "Mechanism of Fullerene Synthesis in the Arc Reactor", *Chemical Physics Letters*, V.286, N 5/6, p.479-485 (1998). A.V.Krestinin, A.P.Moravsky.
- "On the Role of Carbon-Containing Supports in the Stereospecific Propylene Polymerization", *Polymery*, V.43, No.1, p.24-27 (1998). N.M.Galashina, A.N.Klyamkina, N.Yu.Lobanova, G.A.Vinogradov, and A.P.Moravsky.

- "Application of Fullerenes as Catalysts in Chemical Industry", 1st Int. Workshop "Results of Fundamental Research for Investments", S.-Petersburg, Abstr. Invited Lectures, 1998, p.14. A.P.Moravsky.
100. "Modelling the Arc Reactor Process of Fullerene Synthesis", *Molecular Materials*, v.10, 65-70 (1998). A.V.Krestinin, A.P.Moravsky.
- "An Invariant of Carbon Arc Synthesis of Fullerenes", *Fullerene Science and Technology*, v.6, N3, 453-467 (1998). A.P.Moravsky, A.G.Ryabenko, P.V.Fursikov, and A.A.Ryabenko.
- "Composition of Toluene Extracts of Carbon Arc Generated Fullerene Soots", *Molecular Materials*, v.10, 87-92 (1998). A.P.Moravsky, A.G.Ryabenko, P.V.Fursikov, and A.A.Ryabenko.
- "Electrochemical Properties of Pyrolysed Compositions of Polyacrylonitrile and Fullerenes", *Journal of Physics and Chemistry of Solids*, 1998. G.P.Karpacheva, L.M.Zemtsov, V.V.Kozlov, O.N.Efimov, E.P.Krinichnaya, and A.P.Moravsky.
- "The Synthesis and Study of Composites Based on Thermostructured Polyacrylonitrile and Fullerenes", *Molecular Materials*, v.10, 112-114 (1998). G.P.Karpacheva, L.M.Zemtsov, Yu.M.Shulga, O.N.Efimov, E.P.Krinichnaya, A.P.Moravsky, and V.V.Kozlov.
- "Electrochemical Behavior of Polymer Compositions Containing Fullerenes or Nanotubes", *Molecular Materials*, v.11, 107-110 (1998). L.M.Zemtsov, G.P.Karpacheva, V.V.Kozlov, E.P.Krinichnaya, O.N.Efimov, and A.P.Moravsky.
- "Spectrophotometric Determination of the Yield of the C₆₀ and C₇₀ Fullerenes in Electric Arc Synthesis in Helium", *Journal of Analytical Chemistry*, v.53, No.12, 1135-1142 (1998), A.P.Moravskii, A.A.Ryabenko, A.G.Ryabenko, and P.V.Fursikov.
- "A Kinetic Model of Formation of Fullerenes C₆₀ and C₇₀ in Condensation of Carbon Vapor", *Chemical Physics Reports*, v.17, No.9, pp.1687-1707 (1998). A.V.Krestinin, A.P.Moravskii, and P.A.Tesner.
- "Kinetics of Fullerene C₆₀ and C₇₀ Formation in a Reactor with Graphite Rods Evaporated in Electric Arc", *Chemical Physics Reports*, v.18, No.3, pp.515-532 (1999). A.V.Krestinin, A.P.Moravskii.
- "Promotion of Fullerene Hydride Synthesis by Intermetallic Compounds". *Hydrogen Energy Progress XII. Proc. 12th World Hydrogen Energy Conf.* Buenos Aires, Argentina. 1998, v.2, 1221-1230. B.P.Tarasov, V.N.Fokin, A.P.Moravsky, Yu.M.Shulga, V.A.Yartys, and D.V.Schur.
110. "Synthesis of Fullerene Hydrides by Hydrogenation of Fullerite with Hydrogen Evolved from Hydrides of Intermetallic Compounds", *Zh.obshch.khimii*, v.68, N10, p.1585 (1998). B.P.Tarasov, V.N.Fokin, E.E.Fokina, Z.A.Rumynskaya, L.S.Volkova, A.P.Moravskii, Yu.M.Shulga.
- "Synthesis of Crystalline Fullerene Hydrides". *Russ. Chem. Bull.*, N10, 2093-2096 (1998). B.P.Tarasov, V.N.Fokin, A.P.Moravsky, Yu.M.Shulga.
- "Synthesis And Properties of Crystalline Fullerene Hydrides", *NANO-98 (4th Int. Conf. on Nanostructured Materials, Stockholm, Sweden, p.27(1998).* B.P.Tarasov, V.N.Fokin, A.P.Moravsky, Yu.M.Shulga, V.A.Yartys, and D.V.Schur.
- "Hydrogen Sorption-Desorption in Fullerene-Metal(Intermetallics) Systems". *XII Int.Symp. Metal Hydrogen Systems: Fundamentals & Applications (ICHU'98), Hangzhou, China. : Abstracts. 1998, A4:14-P.* B.P.Tarasov, V.N.Fokin, A.P.Moravsky, Yu.M.Shulga, D.V.Schur, and V.A.Yartys.
- "Study of Fullerene-Metals-Hydrogen and Fullerene-Hydrogen Systems". *1998 Autumn Meeting SF2M Metal Hydrides, Paris, Abstracts. 1998, p.31.* B.P.Tarasov, V.N.Fokin, A.P.Moravsky, Yu.M.Shulga, V.A.Yartys, and D.V.Schur.
- "Fullerene Hydrides: Synthesis and Properties". *II Int.Conf."Hydrogen treatment of metals" (HTM-2), Donetsk, Abstracts, p.34 (1998).* B.P.Tarasov, V.N.Fokin, E.E.Fokina, Z.A.Rumynskaya, L.S.Volkova, A.P.Moravskii, and Yu.M.Shulga.
- "Electrochemical Polymerisation of Fullerene C₆₀", *ICSM'98, Abstracts, France, p.45, 1998.* E.P.Krinichnaya, L.I.Tkachenko, O.N.Efimov, A.P.Moravskii, and V.V.Strelets.
- "Soot Aerosol and Fullerene Formation in Carbon Vapor Condensation Process", *4th Int.Symp.Aerosol Soc., S.Petersburg, Abstracts, p32, 1998.* A.V.Krestinin, A.P.Moravsky, P.A.Tesner, and P.V.Fursikov.
- "Vibrational Spectra of C₆₀ Hydrofullerite Prepared Under High Hydrogen Pressure", *Molecular Materials*, v.10, 265-268(1998), I.O.Bashkin, A.I.Kolesnikov, V.E.Antonov, E.G.Ponyatovsky, A.P.Kobzev, A.Yu.Muzychka, A.P.Moravsky, F.E.Wagner, and G.Grosse.
- "Interaction of Platinum Fulleride C₆₀Pt with Deuterium Gas". *Russian Chemical Bulletin*, v.48, No.5, 999 (1999). N.F.Goldshleger, B.P.Tarasov, Yu.M.Shulga, A.A.Perov, O.S.Roshchupkina, and A.P.Moravskii.
120. "Interaction of Platinum Fulleride C₆₀Pt with Deuterium: IR and XPS Studies", In: *"Fullerenes. Recent Advances in the Chemistry and Physics of Fullerenes and Related Materials"*, v.7, eds. K.M.Kadish, P.V.Kamat and D.M.Guldi. The Electrochemical Society, Pennington, NJ, 1999, p.444-449. N.F.Goldshleger, B.P.Tarasov, Yu.M.Shulga, O.S.Roshchupkina, A.A.Perov, and A.P.Moravsky.
- "Layered Materials by Electron Transfer Induced Deposition of Fullerene C₆₀", In: *"Fullerenes. Recent Advances in the Chemistry and Physics of Fullerenes and Related Materials"*, v.7, eds. K.M.Kadish, P.V.Kamat and D.M.Guldi. The Electrochemical Society, Pennington, NJ, 1999, p.444-449. Yu.A.Dubitsky, O.N.Efimov, Yu.V.Korneenkov, E.P.Krinichnaya, A.S.Lobach, A.P.Moravskii, V.V.Strelets, L.I.Tkachenko, and A.Zaopo.
- "SEM and HREM Study of Nanotube Rich Carbon Arc Cathode Deposits Internal Structure", *Carbon*, v.37, pp.1093-1103 (1999). N.A.Kiselev, A.P.Moravsky, A.B.Ormont, and D.N.Zakharov.
- "Neutron Spectroscopy of Fullerite Hydrogenated Under High Pressures". *Physica B: Condensed Matter*, v. 263-264, pp.436-8 (1999). A.I.Kolesnikov, V.E.Antonov, I.O.Bashkin, J.C.Li, A.P.Moravsky, E.G.Ponyatovsky and J.Tomkinson..

- "Inelastic and Compton Neutron Scattering Study of Pristine Fullerite, Polymerized Fullerite and Hydrofullerite", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.22. I.O.Bashkin, V.E.Antonov, A.I.Kolesnikov, J. Mayers, A.P.Moravsky, S.F.Parker, E.G.Ponyatovsky and J.Tomkinson..
- "Magnetic Properties of High-Pressure Hydrogenated and Polymeric Fullerenes", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p. 167. I.O. Bashkin, V.E. Antonov, A.P. Moravsky, Yu.G. Morozov, Yu.M. Shulga, Yu.A. Ossipyan and E.G. Ponyatovsky.
- "Magnetic Properties of High-Pressure Hydrogenated and Polymeric Fullerenes", Mol. Materials, v.13, No.1-4, 263-268 (2000). I.O. Bashkin, V.E. Antonov, Yu.A. Ossipyan, E.G. Ponyatovsky, A.P. Moravsky, Yu.M. Shulga and Yu.G. Morozov.
- "A Dissolution-Precipitation Model for the Catalyst of SWNT Growth", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.109. R.O.Loutfy, T.P.Lowe, J.L.Hutchison, N.A.Kiselev, D.N.Zakharov, A.V.Krestinin, E.P.Krinichnaya and A.P.Moravsky.
- "A Parametric Study of Co/Ni Catalyst for SWNT Synthesis", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.117, R.O.Loutfy, T.P.Lowe, J.L.Hutchison, N.A.Kiselev, D.N.Zakharov, E.P.Krinichnaya, V.E.Muradyan, B.P.Tarasov and A.P.Moravsky.
130. "Hydrogen Storage on Fullerenes: Catalytic Approach and Analysis", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.226. R.O.Loutfy, E.M.Veksler, and A.P.Moravsky.
- "Fullerene Black as a Catalyst for n-Alkane Conversions", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.230. P.V.Fursikov, S.D.Kushch, V.E.Muradyan, G.I.Davydova, E.I.Knerelman and A.P.Moravsky.
- "Platinum Fulleriide Reduction by Molecular Deuterium or 9,10-Dihydroanthracene", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.255. N.F.Goldshleger, B.P.Tarasov, Yu.M.Shulga, O.S.Roschupkina, A.A.Perov, A.V.Okotrub and A.P.Moravsky.
- "Investigation of Fullerite-Deuterium Systems", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.256. B.P. Tarasov, Yu.M.Shulga, V.N.Fokin, E.E.Fokina, A.P.Moravsky, D.V.Schur, S.Yu.Zaginaichenko and V.A.Yartys.
- "Electrosynthesis of Fullerene Coatings on Carbon Fibres", 4th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 1999, p.301. E.P.Krinichnaya, A.P.Moravsky, V.V.Strelets and O.N.Efimov.
- "Carbon Nanotubes Production and Applications", 1999 US Army Sagamore Conference, Sagamore, Abstracts, p.26 (1999), R.O.Loutfy, A.P.Moravsky, T.P.Lowe, and J.C.Withers.
- "Persistent Currents and Magnetic Flux Trapping in Fragments of Carbon Deposits Containing Multiwalled Nanotubes", JETP Letters., v.70, No.7, 462-468 (1999), V.I.Tsebro, O.E.Omelyanovskii, and A.P.Moravsky.
- "Process for Producing a Superconductive Layered Material and Product Obtainable Therefrom", European Patent DC98039 (1999), Yu.A.Dubitsky, A.Zaopo, O.N.Efimov, and A.P.Moravsky.
- "Scaled-Up Production of Carbon Nanotubes", Taiwan-Russian Hi-Tech Forum, Taipei, July 2000, Ext.Abstr., pp.51-73 (2000). A.P.Moravsky.
- "Magnetic Properties of High Pressure Hydrogenated and Polymeric Fullerenes", Molecular Materials, v.13, No.1-4, 263-268 (2000), I.O.Bashkin, V.E.Antonov, Yu.A. Ossipyan, E.G.Ponyatovsky, A.P.Moravsky, Yu.M. Shulga, and Yu.G. Morozov.
140. "Hydrogen in the Vibrational Spectra of High-Pressure Hydrofullerite", Molecular Materials, v.13, No.1-4, 251-256 (2000). I.O.Bashkin, V.E.Antonov, A.I.Kolesnikov, E.G.Ponyatovsky, J.Mayers, S.F.Parker, J.Tomkinson, A.P.Moravsky, and Yu.M.Shulga.
- "Fullerene Black as a Catalyst for n-Alkane Conversions", Molecular Materials, v.13, No.1-4, 319-324(2000). P.V.Fursikov, S.D.Kushch, V.E.Muradyan, G.I.Davydova, E.I.Knerelman, and A.P.Moravsky.
- "Interaction of Hydrogen with Single-Walled Carbon Nanotubes Under High Pressure", Int.J. Alternative Energetics and Ecology, v.1, 134 (2000). I.O.Bashkin, V.E.Antonov, O.V.Boltalina, I.V.Goldt, A.F.Gurov, A.I.Kolesnikov, E.P.Krinichnaya, A.P.Moravsky, S.S.Khasanov, Yu.M.Shulga, and E.G.Ponyatovsky.
- "Hydrogen Absorption by C₆₀ and Carbon Nanotubes under High Pressure", 1st Int. Seminar on Safety and Economics of Hydrogen Transport, July 25-28, 2000, Sarov, Russia, Abstracts, p.34 (2000). I.O.Bashkin, V.E.Antonov, O.V.Boltalina, I.V.Goldt, A.F.Gurov, A.I.Kolesnikov, E.P.Krinichnaya, A.P.Moravsky, S.S.Khasanov, Yu.M.Shulga, and E.G.Ponyatovsky.
- "Carbon Nanotubes and Nanofibres: Synthesis, Properties and Prospects for Applications", Russ.Conf. on Nanochemistry and Nanoparticles, Moscow, Abstr., p.5 (2000). B.P. Tarasov, Yu.M. Shulga, V.E. Muradyan, E.I. Krinichnaya, G.I. Zvereva, P.V.Fursikov, S.D.Kushch, O.V.Golodkov, A.G.Ryabenko, A.V.Krestinin, A.P.Moravsky, and O.N.Efimov.
- "Graphite Oxide-based Nickel-Containing Catalysts for Reductive Dechlorination of Polychlorinated Aromatic Hydrocarbons", Russian Chemical Bulletin, No.6, 1023-1025 (2000). V.E.Muradyan, V.S.Romanova, A.P.Moravsky, Z.N.Parnes, and Yu.N.Novikov.
- "Layered Materials by Electrochemical Deposition of Alkali Fullerenes", Fullerene.Science and Technology, v.8, 17-25 (2000), Yu.A.Dubitsky, A.Zaopo, O.N.Efimov, Yu.V.Korneenkov, E.P.Krinichnaya, A.P.Moravsky, V.V.Strelets, and L.I.Tkachenko
- "Carbon-Based Catalyst for C-C and C-H Bonds Activation", Russ.Patent (Pending), 2000. N.I.Burangulov, A.P.Moravsky.
- "Fullerene-Based Filter for Cigaretts", Russ.Patent Appl. 011045 (2000). N.I.Burangulov, A.P.Moravsky, P.A.Pogorelyi.

- "Fullerene-Based Adsorbent for Clearing and Purification of Alcohol and Other Liquids", Russ. Patent Appl. 011046 (2000). N.I. Burangulov, V.N. Knyazev, V.V. Nesterov, A.P. Moravsky, and V.V. Sulima.
- "Cosmetic Cream", Russ. Patent Appl. 011047 (2000). N.I. Burangulov, G.I. Dyachuk, V.N. Zgonnik, G.I. Mamleeva, A.P. Moravsky, P.A. Pogorelyi.
150. "Double-Walled Carbon Nanotubes and Methods of Production and Application", US Patent (Pending), 2000. A.P. Moravsky, R.O. Loutfy.
- "Use of Fullerenes and Fullerene-Containing Materials in Catalysis: A Review", *Petroleum Chemistry*, v.40, 365-377 (2000), N.F. Goldshleger and A.P. Moravsky.
- "Hydrogenation of Fullerite under High Hydrogen Pressures", Russ. Conf. "Phase Transitions at High Pressures", Abstracts, Chernogolovka, 2000, p.13/2. I.O. Bashkin, V.E. Antonov, A.F. Gurov, A.I. Kolesnikov, J. Meiers, A.P. Moravsky, C.F. Parker, E.G. Ponyatovsky, S.S. Khasanov, Yu.M. Shulga.
- "Hydrogen Adsorption by Carbon Nanotubes Under 30 kbar Hydrogen Pressure", Russ. Conf. "Phase Transitions at High Pressures", Abstracts, Chernogolovka, 2000, p.13/4. I.O. Bashkin, V.E. Antonov, A.F. Gurov, E.P. Krinichnaya, A.P. Moravsky, E.D. Obratsova and E.G. Ponyatovsky.
- "Vibrational Spectra of C_{60} Polymer Phases Obtained Under Pressures to 55 kbar", Russ. Conf. "Phase Transitions at High Pressures", Abstracts, Chernogolovka, 2000, p.13/3. I.O. Bashkin, A.I. Kolesnikov, J.-Ch. Li, A.P. Moravsky, C.F. Parker, E.G. Ponyatovsky.
- "Magnetic Ordering in Hydrofullerites Saturated with Hydrogen at High Pressures", Russ. Conf. "Phase Transitions at High Pressures", Abstracts, Chernogolovka, 2000, p. Y/2. V.E. Antonov, I.O. Bashkin, A.P. Moravsky, Yu.G. Morozov, Yu.A. Ossipyan, E.G. Ponyatovsky, S.S. Khasanov, Yu.M. Shulga.
- "Magnetic Ordering in Hydrofullerite $C_{60}H_{24}$ ", Int. Symp. Metal-Hydrogen Systems: Fundamentals and Applications, October 1-6, 2000, Noosa, Queensland, Australia, Abstracts, p.56 (2000). V.E. Antonov, I.O. Bashkin, S.S. Khasanov, A.P. Moravsky, Yu.G. Morozov, Yu.M. Shulga, Yu.A. Ossipyan, and E.G. Ponyatovsky.
- "Production and Characterization of Fullerene Hydrides", Ch. in: *Encyclopaedia of Materials*, Japan, 2001, R.O. Loutfy, A.P. Moravsky, E.M. Wexler.
- "Dehydrogenation and Aromatization of Methane over Fullerene Catalysts", *Neftekhimiya*, (2001). P.V. Fursikov, S.D. Kushch, V.E. Muradian, Yu.V. Butenko, E.I. Knerelman, E.V. Koldasheva, V.L. Kuznetsov, A.P. Khrushch, A.P. Moravsky.
- "Double-Walled Carbon Nanotubes Fabricated by Hydrogen Arc Discharge Method", *Carbon*, v.39, 761-770 (2001), J.L. Hutchison, N.A. Kiselev, E.P. Krinichnaya, A.V. Krestinin, R.O. Loutfy, A.P. Moravsky, V.E. Muradyan, E.D. Obratsova, J. Sloan, S.V. Terekhov, D.N. Zakharov.
- 160 "Prospects of Fullerene Commercial Applications", Abstr. Int. Fullerene Workshop 2001, Tokyo, Japan, Mitsubishi Corp., p.47 (2001). R.O. Loutfy, J.C. Withers, T. Lowe, A.P. Moravsky, S. Dimitrievich, E. Veksler, M. Hecht, and A. Kolesnikov.
- "Commercial Production of Fullerenes and Carbon Nanotubes", Proc. Int. Fullerene Workshop 2001, E. Osawa (Ed), Tokyo, Japan, Mitsubishi Corp., (2001), in the press. R.O. Loutfy, T.P. Lowe, A.P. Moravsky, and S. Katagiri.
- "Principal Component Analysis of Fullerenes C_{76} , C_{78} and C_{84} Content Variability in Carbon Arc Soot Extracts", 5th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 2001, p.111. A.P. Moravsky, A.G. Ryabenko, P.V. Fursikov, and A.A. Ryabenko.
- "Magnetic Properties of Carbon Nanotubes Produced by Arc-Discharge Method at Different Conditions", 5th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 2001, p.126. A.S. Kotosonov, D.V. Shilo, and A.P. Moravsky.
- "Using Absorption Spectroscopy for Determination of SWNT", 5th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 2001, p.205. A.G. Ryabenko, P.V. Fursikov, E.P. Krinichnaya, A.P. Moravsky, T.V. Dorofeeva, and G.I. Zvereva.
- "BCC and FCC C_{60} Hydrofullerites after High-Pressure Synthesis", 5th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 2001, p.241. I.O. Bashkin, V.E. Antonov, A.I. Harkunov, A.I. Izotov, S.S. Khasanov, V.I. Kulakov, A.P. Moravsky, Yu.A. Ossipyan, E.G. Ponyatovsky, Yu.M. Shulga, E.A. Shteinman, and L.V. Zorina.
- "Fullerenes under High Hydrogen Pressure", VII Int. Conf. on Hydrogen Materials Science and Chemistry of Metal Hydrides (ICHMS'01), Sep. 16-22, Alushta, Ukraine, Abstracts, p. 686-7 (2001). I.O. Bashkin, G.E. Abrosimova, V.E. Antonov, A.I. Harkunov, A.N. Izotov, S.S. Khasanov, V.I. Kulakov, A.P. Moravsky, Yu.A. Ossipyan, E.G. Ponyatovsky, Yu.M. Shulga, E.A. Shteinman, and L.V. Zorina.
- "XRD Studies of Hydrofullerites Produced under High Hydrogen Pressure", 3rd Natl. Conf. on Application of X-Ray, Synchrotron, Neutron and Electron Spectroscopies for Materials Studies (RSNA), May 21, Moscow, Inst. Cryst. RAN, Abstracts, p.48 (2001). I.O. Bashkin, V.E. Antonov, L.V. Zorina, A.I. Izotov, V.I. Kulakov, A.P. Moravsky, Yu.A. Ossipyan, E.G. Ponyatovsky, A.I. Harkunov, S.S. Khasanov, E.A. Shteinman, Yu.M. Shulga.
- "Study of Porous Structure, Density of Adsorbed Hydrogen in Carbon Nanomaterials and Its Electrochemical Generation, Adsorption-Desorption and Electrooxidation in These Materials", 5th Int. Workshop Fullerenes & Atomic Clusters, St. Petersburg, Russia, Book of Abstracts, 2001, p.286. Yu.M. Volkovich, O.N. Efimov, B.P. Tarasov, A.Yu. Rychagov, E.P. Krinichnaya, V.E. Sosenkin, N.F. Nikolskaya, R.O. Loutfy, and A.P. Moravsky.
- "Studies of Porous Structure and Electrochemical and Electroadsorption Properties of Carbon Nanotubes and Nanofibers", Abstr. 7th Int. Conf. Hydrogen Materials Science and Chemistry of Metal Hydrides (ICHMS'01), Crimea, Ukraine, 2001, pp.32-33. Yu.M. Volkovich, O.N. Efimov, B.P. Tarasov, A.Yu. Rychagov, E.P. Krinichnaya, V.E. Sosenkin, N.F. Nikolskaya, R.O. Loutfy, and A.P. Moravsky.
- 170 "Electrochemical generation and oxidation of hydrogen in carbon nanomaterials of attested porous structure", Proceedings of International Workshop on Science and Application of Nanotubes, Germany, 22-25, 2001, Yu.M. Volkovich, O.N. Efimov, B.P. Tarasov, A.Yu. Rychagov, E.P. Krinichnaya, V.E. Sosenkin, N.F. Nikolskaya, R.O. Loutfy, A.P. Moravsky.
- "Fullerenes and Fullerene-Containing Materials in Catalysis", *Fullerene Science and Technology*, No.3, 255 (2001). N.F. Goldshleger, and A.P. Moravsky.

- "Hydrogen-Containing Carbon Nanostructures: Synthesis and Properties" (Review), Russian Chemical Reviews, v.70, No.2, 131-146 (2001), B.P.Tarasov, N.F.Goldshleger, and A.P.Moravsky.
- "ESR Study of Nanotubes and Nanoparticles Produced by Carbon Arc Technique under Various Helium Pressures", J.Experim.Theoret.Physics (Russ), 2001, in the press, A.S.Kotosonov, D.V.Shilo, A.P.Moravsky, and N.A.Kiselev.
- "Platinum Fulleride Reduction by Molecular Deuterium or 9,10-Dihydroanthracene", Fullerene Science and Technology, v.8, No.6, pp. 519-529 (2000), N.F.Goldshleger, B.P.Tarasov, Yu.M.Shulga, O.S.Roschupkina, A.A.Perov, A.V.Okotrub, and A.P.Moravsky.
- "Microwave Properties of Fullerene Black as Dependent of Conditions for Its Synthesis", Khimiya Vysokikh Energii, (Russ), 2001, in the press, E.A.Sokolov, S.D.Babenko, D.N.Zakharov, A.P.Moravskii, N.P.Piven, B.P.Tarasov, and P.V.Fursikov.
- "Field Emission Display with Double-Walled Carbon Nanotube Emitters", Proceedings of 21st International Display Conference/ 8th International Display Workshops; 2001; pp.1245-1248, H.Kurachi, S.Uemura, J.Yotani, T.Nagasako, H.Yamada, T.Ezaki, T.Maesoba, R.Loutfy, A.Moravsky, T.Nakazawa, S.Katagiri, and Y.Saito.
- "Field Emission from Double-Walled Carbon Nanotube Cathodes", Asia Display/ IDW'01, Proceedings of 21st International Display Research Conference/ 8th International Display Workshops; 2001; pp. 1237-1240, H.Kurachi, S.Uemura, J.Yotani, T.Nagasako, H.Yamada, T.Ezaki, T.Maesoba, R.Loutfy, A.Moravsky, T.Nakazawa, S.Katagiri, and Y.Saito.
- "Electric Arc Synthesis of Carbon Nanotubes", Proceedings of the Conf. "Hydrogen Material Science and Chemistry of Metal Hydrides", Kiev, ADEF, 2001, 548-551. V.E.Muradyan, B.P.Tarasov, Yu.M.Shul'ga, A.G.Ryabenko, P.V.Fursikov, N.S.Kuyunko, A.P.Moravsky, S.V.Terekhov, S.N.Bokova, E.D.Obraztsova, S.Yu.Zaginaichenko, D.V.Shchur.
- "Physical Hydrogen Storage on Nanotubes and Nanocarbon Materials", Perspectives of Fullerene Nanotechnology (Proc. Int.Fullerene Workshop 2001, Tokyo, Japan, Mitsubishi Corp.), E.Osawa (Ed), Kluwer AP, Dordrecht, 2002, pp. 327-340, R.O.Loutfy, A.P.Moravsky, A.Franco, and E.M.Wexler.
180. "Nanotubes as Anode Material For Lithium-Ion Batteries", Perspectives of Fullerene Nanotechnology (Proc. Int.Fullerene Workshop 2001, Tokyo, Japan, Mitsubishi Corp.), E.Osawa (Ed), Kluwer AP, Dordrecht, 2002, pp. 341-356, R.O.Loutfy, S.Hossain, A.P.Moravsky, and M.Saleh.
- "Commercial Production of Fullerenes and Carbon Nanotubes", Perspectives of Fullerene Nanotechnology (Proc. Int.Fullerene Workshop 2001, Tokyo, Japan, Mitsubishi Corp.), E.Osawa (Ed), Kluwer AP, Dordrecht, 2002, pp. 35-48, R.O.Loutfy, T.P.Lowe, A.P.Moravsky and S. Katagiri.
- "Magnetic Properties of Carbon Nanotubes Produced by the Arc-Discharge Method under Different Conditions", Physics of Solid State, vol.44, No.4, 666-667 (2002), A.S.Kotosonov, D.V. Shilo, A.P. Moravskii.
- "Magnetic Ordering in Hydrofullerite C₆₀H₂₄", Journal of Alloys and Compounds, No. 330-332, 365-368 (2002). V.E.Antonov, I.O.Bashkin, S.S.Khasanov, A.P.Moravsky, Yu.G.Morozov, Yu.M.Shulga, Yu.A.Ossipyan, and E.G.Ponyatovsky.
- "Optical Properties of Carbon Nanotubes Filled with Fullerenes and Their Derivatives", 1st Seminar on Carbon Nanotechnologies, Abstracts, Moscow, 2002. A.G.Ryabenko, T.V.Dorofeeva, A.P.Moravsky, V.V.Roddatis, N.A.Kiselev.
- "Field Emission Properties of Carbon Nanotubes Grown on Various Metal Substrates", 15th Int. Vacuum Microelectronics Conf. and 48th Int. Field Emission Symp., IVMC & IFES 2002, Lyon, France, July 7-11, Ext.Abst. (2002). A.G. Chakhovskoi, N.N. Chubun, C.E.Hunt, R.O.Loutfy, A.P.Moravsky.
- "Field Emission Characterization of Carbon Nanotubes Grown on Metal Substrates", Int. Conf. " ", Austin, TX, (2003). A. Chakhovskoi, N. Chubun, R.Loutfy, A.Moravsky
- "Isomer Structure of High-Pressure Hydrofullerene Probed by Soft X-Ray Emission", Journal of Molecular Structure (Theochem), v.639, No.1-3, 27-33 (2003). E.Z.Kurmaev, A.Moeves, T.Ida, S.Danielache, K.Endo, I.O.Bashkin, A.I.Harkunov and A.P.Moravsky.
- "Thermally Stable Hydrogen Compounds of Carbon Nanotubes and Nanofibers Obtained Under High Pressure", JETP Letters, v.79, 280-285 (2004). I.O.Bashkin, V.E.Antonov, A.V.Bazhenov, I.L.Bdikin, D.N.Borisenko, E.P.Krinichnaya, A.P.Moravsky, A.I.Kharkunov, Yu.M.Shulga, Yu.A.Ossipyan, and E.G.Ponyatovsky.
- "Anomalous Soft Dynamics of Water in a Nanotube: A Revelation of Nanoscale Confinement", Phys. Rev. Lett., v.93, 035503-035507 (2004), A.I.Kolesnikov, J.-M.Zanotti, C.-K.Loong, P.Thiyagarajan, A.P.Moravsky, R.O.Loutfy, C.J.Burnham.
190. "Anomalous Soft Dynamics of Water in a Nanotube: A Revelation of Nanoscale Confinement", Virtual J. Nanoscale Science and Technology, <http://www.vjnano.org>, July 26 (2004), A.I.Kolesnikov, J.-M.Zanotti, C.-K.Loong, P.Thiyagarajan, A.P.Moravsky, R.O.Loutfy, C.J.Burnham.
- "Carbon Nanotubes Full of Water", Chemical & Engineering News, v.82, No.26, p.13 (2004). M.Jacoby: A.I.Kolesnikov, J.-M.Zanotti, C.-K.Loong, P.Thiyagarajan, A.P.Moravsky, R.O.Loutfy, C.J.Burnham.
- "Small Ice", Ice and Icy Particles Workshop, Telluride, Yuly 2004, p.13. C.J.Burnham, A.I.Kolesnikov, J.-M.Zanotti, C.-K.Loong, P.Thiyagarajan, A.P.Moravsky, R.O.Loutfy.
- "Mechanism of Carbon Transfer in the Arc Synthesis of DWNTs", 5th Int.Conf.Sci.Appl.Nanotubes NT'04, Mexico, Abstr., p.62 (2004). A.P.Moravsky, R.O.Loutfy.
- "Carbon Nanotubes as Thermionic Emitters", 5th Int.Conf.Sci.Appl.Nanotubes NT'04, Mexico, Abstr., p.217 (2004). R.O.Loutfy, M.Samandi, A.P.Moravsky, S.Strange.

- "Composition of the Extracts of Fullerene Soot Obtained in the Arc Discharge Reactor", Zh. Fiz. Khimii, v.78, No.4, 1-8 (2004). A.G.Ryabenko, V.I.Kozlovskii, A.P.Moravsky, A.A.Ryabenko, P.V.Fursikov.
- "Mass-Spectral Study of Fullerenes C₇₆ and C₈₄ Evolution in the Arc Synthesis", Zh. Fiz. Khimii, (2004). A.G.Ryabenko, V.I.Kozlovskii, A.P.Moravsky, A.A.Ryabenko, P.V.Fursikov.
- "Laser Desorption and Electro-Spray TOF MS Studies of Hot Fullerenes C₇₈ and C₈₄ Reactions", Mass-Spectroscopiya, v.1, No.2, 135-142 (2004). V.I.Kozlovskii, A.G.Ryabenko, A.P.Moravsky, M.F. Budyka.
- "Raman Studies of Natural and Synthetic Carbons", VII Conf. "Analitica Sibiri i Dalnego Vostoka", Oct.11, 2004, Abstr., Novosibirsk, 12 (2004), T.N.Moroz, A.G.Ryabenko, V.G.Kostrovskii, S.M.Zhmodik, A.G.Mironov, A.P.Moravsky, A.M.Spiridonov.
- "Carbon Micro- and Nanotubes synthesized by PE-CVD Technique: Tube Structure and Catalytic Particles Crystallography", Carbon, V.42, No.1, 149-161 (2004). N.A.Kiselev, J.L.Hutchison, A.P.Moravsky, E.V.Rakova, E.V.Dreval, C.J.D. Hetherington, D.N.Zakharov, J.Sloan, R.O.Loutfy.
200. "Spectrophotometry and Electron Microscopy of SWNTs filled with C₆₀, C₇₀ and C₆₀H₂₈ molecules", Conf. "Nanodimensional Systems (NANSYS)", Oct.12, 2004., Kiev, A.G.Ryabenko, N.A.Kiselev, J.L.Hutchison, A.P.Moravsky
- "Studies of Internal Fillers Influence on Spectral Properties of SWNTs", 3rd Int. Conf. "Carbon: Fundamental Problems of Science, Materials and Technology", Oct.13-15, Moscow, Abstracts, p.197 (2004). A.G.Ryabenko, T.N.Moroz, V.G.Kostrovskiy, S.S.Bukalov, N.A.Kiselev, J.L.Hutchison, A.P.Moravsky
- "Electrochemical Polymerization of C₆₀ Induced by Superoxide Radical Anion, O₂⁻", Int. Society of Electrochemistry, 55th Annual Meeting, Thessaloniki, Greece, 19-24 September 2004, paper S10FP60. E.P.Krinichnaya, A.P.Moravsky, O.Efimov, K.Winkler, J.W.Sobczak, W.Kutner, A.L.Balch.
- "Mechanistic Studies of the Electrochemical Polymerization of C₆₀ in the Presence of Dioxygen or C₆₀O", Abstracts of the 10th Int. Conf. on Electroanalysis of the European Society for ElectroAnalytical Chemistry, ESEAC 2004, Galway, Ireland, June 6-10, 2004, p.123. Elena P. Krinichnaya, Alexander P. Moravsky, Oleg Efimov, Janusz W. Sobczak, Krzysztof Winkler, Włodzimierz Kutner, and Alan L. Balch.
- "Mechanistic Studies of the Electrochemical Polymerization of C₆₀ in the Presence of Dioxygen or C₆₀O", Journal of Materials Chemistry, 2005, v.15, 1468-1476, E.P.Krinichnaya, A.P.Moravsky, O.Efimov, Ja.W. Sobczak, K.Winkler, W.Kutner, A.L.Balch.
- "Neutron Spectroscopy of Carbon Nanomaterials". Proc. MRS Fall Meeting, Symp.Q, paper Q2.3, 2004. A.I. Kolesnikov, C.-K. Loong, A.P. Moravsky, R.O. Loutfy, C.J.Burnham.
- "Inelastic neutron scattering study of carbon nano-materials", 8th Int. Conf.on Applications of Diamond and Related Materials/1st NanoCarbon Joint Conference, Argonne National Laboratory, Abstract p.123, May 15-19, 2005. A.I. Kolesnikov, C.-K. Loong, A.P. Moravsky, and R.O. Loutfy.
- "Anomalous Soft Dynamics of Water in Carbon Nanotubes", International Conference on Neutron Scattering ICNS2005, Abstr.No 326, 2005, pp.101-103, A.I.Kolesnikov, C.-K.Loong, N.R. de Souza, C.J.Burnham, A.P.Moravsky.
- "Growth of Carbon Nanotubes by Arc Discharge and Laser Ablation", Ch.3 in the book "Carbon Nanotube Science and Applications", CRC Press, Ed. M.Meyyappan, 2005, pp.65-97. A.P.Moravsky, E.M.Wexler, R.O.Loutfy.
- "Mechanism of the Arc Synthesis of DWNTs", 2nd NASA/Rice University Workshop on SWNT Nucleation and Growth Mechanism, Guadalupe River Ranch, Boerne, Texas, 8-12 April 2005, Abstracts, p.53. A.P.Moravsky, R.O.Loutfy, T.P.Lowe.
210. "Hydrogenated Single-Walled Carbon Nanotube Material as a Cold-Neutron Moderating Medium", Proceedings of ICANS-XVII, 17-th Meeting of the International Collaboration on Advanced Neutron Sources, April 25-29, 2005, Santa Fe, New Mexico, p. 311-317. C.-K.Loong, A.I.Kolesnikov, N.R. de Souza, J.M.Carpenter, I.O.Bashkin, I.V.Kondratieva, L.H.He and A.P.Moravsky.
- "Fluid Ice in Carbon Nanotubes", 6th Int.Conf.Sci.Appl.Nanotubes NT'05, Sweden, Abstr., p.66 (2005). A.I.Kolesnikov, C.J.Burnham, C.-K.Loong, N.R. de Souza, A.P.Moravsky and R.O.Loutfy.
- "Neutron Spectroscopy of Water in Carbon Nanotubes", 2nd Workshop on Inelastic Neutron Spectrometers 2005, WINS2005, Abstracts, Dec.5, Cairns, Australia, A.I.Kolesnikov, C.-K.Loong, C.J.Burnham, N.R. de Souza, G.Reiter, J.Mayers, A.P.Moravsky and R.O.Loutfy.
- "Evolved Gas Analysis of Heat-Treated Carbon Nanomaterials", Mater.Res.Soc.Symp.Proc. Vol.885E, pp.0885-A09-34.1 - 0885-A09-34.6 (2006). Yu.M.Shulga, V.M.Martynenko, R.O.Loutfy, A.P.Moravsky.
- "Bulk Production of a Strong Covalently Linked (C₆₀H₂)₂ Dimer", J. Phys. Chem. B., v. 109 (24), 11875-11879 (2005). Yu.V.Vasil'ev, S.G.Kotsiris, I.O.Bashkin, V.E.Antonov, A.P.Moravsky and T.Drewello.
- "High-pressure hydrofullerites: Evidence for a covalently linked C₆₀H₃₆ dimer". MRS Meeting (2005), Boston. Yury V. Vasiliev, Sotirios G. Kotsiris, Igor O. Bashkin, Vladimir E. Antonov, Alexander P. Moravsky and Thomas Drewello.
- "Synthesis of Bamboo-like Nanofibers on Si/TiNx supported Fe-Mo Catalyst", IWFA' 2005, Abstract P252, St.Petersburg, p.317, A.A.Volodin, P.V.Fursikov, B.P.Tarasov, O.N.Efimov, Yu.M.Shul'ga, N.I.Shuvalova, Yu.A.Kasumov, A.A.Pasynsky and A.P.Moravsky.
- "Kinetics of the Arc Synthesis of DWNTs", IWFA' 2005, Abstract P163, St.Petersburg, p.317, A.P.Moravsky, R.O.Loutfy.
220. "Electrochemical Modification of Nanocarbon Coatings", Alternative Energetics and Ecology, vol.1, No.1, 123 (2005). O.N.Efimov, E.P.Krinichnaya, V.V.Strelets, L.I.Tkachenko, E.V.Ovsyannikova, N.M.Alpatova, A.P.Moravsky, R.O.Loutfy.

- "Water in Carbon Nanotubes: Neutron Spectroscopy and MD Simulations", IPNS ANL, 25-th Anniversary Progress Report, May 2006. A.I.Kolesnikov, C.-K.Loong, N.R.deSouza, P.Thiygarajan, C.J.Burnham, A.P.Moravsky, J.-M.Zanotty.
- "Double-Walled Carbon Nanotubes under Hydrostatic Pressure: Raman Experiments and Simulations", J.Nanoscience and Nanotechnology, 2006, V.Gadagkar, S.Saha, D.V.S.Muthu, P.K.Maiti, Y.Lansac, A.Jagota, A.Moravsky, R.O.Loutfy and A.K.Sood.
- "Dynamics of water confined in single- and double-wall carbon nanotubes", J. Chem. Phys., v.124, 194703-1 - 194703-6 (2006). E. Mamontov, C. J. Burnham, S.-H. Chen, A. P. Moravsky, C.-K. Loong, N. R. de Souza, and A. I. Kolesnikov
- "XPS Study of Fluorinated Multi-Walled Carbon Nanotubes", J.Electron Spectroscopy and Related Phenomena, 2006. Y.M. Shulga, Ta-Chang Tien, Chi-Chen Huang, Shen-Chuan Lo, V.E. Muradyan, N.V. Polyakova, Yong-Chien Ling, R.O.Loutfy and A.P.Moravsky.
- "RF Plasma Method for Production of Single Walled Carbon Nanotubes", US Patent No. US 7,052,667 B2 of May 30, 2006, to R.O.Loutfy, A.P.Moravsky, T.P.Low.
- "Double-Walled Carbon Nanotubes and Methods for Production and Application", Korean Patent No. 10-0604459, Registered Jul.18, 2006, Patent allowed May 16, 2006, Appln. No. 7012344/2002, Filed Sep.18, 2002, Priority of Oct. 6, 2000. Issued to: A.P.Moravsky and R.O.Loutfy.
- "Anomalous Soft Dynamics of Water in Carbon Nanotubes", Physica B: Physics of Condensed Matter, v.385-386, 272-274 (2006); A.I.Kolesnikov, C.-K.Loong, N.R. de Souza, C.J.Burnham, A.P.Moravsky.
- "Dielectric Properties of Carbon Nanotube – Epoxy Composites in UHF Range", 4th Int. Conf. "Carbon: Fundamental Problems of Science, Materials and Technology", Oct.13-15, Moscow, Abstracts, p.78 (2006). V.E.Muradyan, E.A.Sokolov, S.D.Babenko, A.P.Moravsky, G.A.Nikolaeva.
- "Anomalous Behavior of Proton Zero Point Motion in Water Confined in Carbon Nanotubes", Phys. Rev. Lett., v.97, 247801-247804 (2006). G. Reiter, C. Burnham, D. Homouz, P.M. Platzman, J. Mayers, T. Abdul-Redah, A.P.Moravsky, J.C. Li, C.-K. Loong and A.I.Kolesnikov.
- 230 "Water in Carbon Nanotubes Is Not the Same Old Stuff", Nature, in press, G.Reiter, D.Homouz, C.Burnham, P.M.Platzman, J.Mayers, T.Abdul-Redah, A.P.Moravsky, J.C.Li, C.-K.Loong, A.Kolesnikov.
- "Dielectric Properties of Carbon NanoFiber Composites in UHF Range", Abstr. 4th Russian Conf. Irreversible Processes in Nature and Technique, Moscow, Jan. 29-31, p.28, (2007). V.E.Muradyan, E.A.Sokolov, S.D.Babenko, A.P.Moravsky, G.A.Nikolaeva.
- "Spectral Properties of Single-Walled Carbon Nanotubes Encapsulating Fullerenes", Carbon v.45, No.7, 1492-1505 (2007). A.G. Ryabenko, N.A. Kiselev, J.L. Hutchison, T.N. Moroz, S.S. Bukalov, L.A. Mikhailitsyn, R.O. Loutfy, A.P. Moravsky.
- "Observation of a Dynamic Crossover in Water Confined in Double-Wall Carbon Nanotubes", Phys.Rev.E, xxx(2007)xxx-xxx. X.-Q. Chu, A. I. Kolesnikov, A. P. Moravsky, V. Garcia-Sakai, and S.-H. Chen.
- "Neutron Spectroscopy Study of Single-Walled Carbon Nanotubes Hydrogenated under High Pressure", J. Alloys and Compounds, v.446-447, 389-392 (2007). A.I.Kolesnikov, I.O.Bashkin, V.E.Antonov, D.Colognesi, J.Mayers, A.P.Moravsky.

Run CF39

 H_2 175 Torr (600 scan) + Ar 125 Torr (200 scan) = 350 TorrRod: $C_{60}(2'')$ I81A @ 2.0 mm/min

$\phi 8.2$ 1.3 $\phi 3.16$

$$\begin{array}{r} 21.81g \\ - 16.66 \\ \hline 5.15g \end{array}$$
 $5.15g (4:1 = M: Graph)$ $\hookrightarrow 4.12g M + 1.03g C$

Chin Cat	51.05% Ni
Composition	13.8 Co
	27.1 Fe
	8.05 S

Steady-state vaporization found at 11.26-28V
at I81 @ 2.0

Composition of the rod:

 $\Sigma C = 16.65 - 1.3 + 1.03 = 16.4g$; $\Sigma C + M = 20.52g$ Chung $Ni = 2.103g \Rightarrow \frac{2.103}{16.4} \cdot \frac{12}{58.69} = 2.62 at\% = 10.26 wt\%$ 2.6 $Co = 4.12 \cdot 0.138 \Rightarrow \frac{4.12}{16.4} \cdot \frac{58.93}{58.69} = 0.706 at\% = 2.77 wt\%$ 0.7 $Fe = 4.12 \cdot 0.271 = 1.116$ 1.46 at% = 5.44 wt% 1.45 $S = 4.12 \cdot 0.0805 = 0.333$ 0.76 at% = 1.62 wt% 0.75

Total burning 45 min

Product: 1.17g WS: pWS 0.45g MG/B: passes sieve

0.72g sieved powder

Deposit + Yuk 12.4g
Sieves 2.8g

Rod Remainder 1.6g

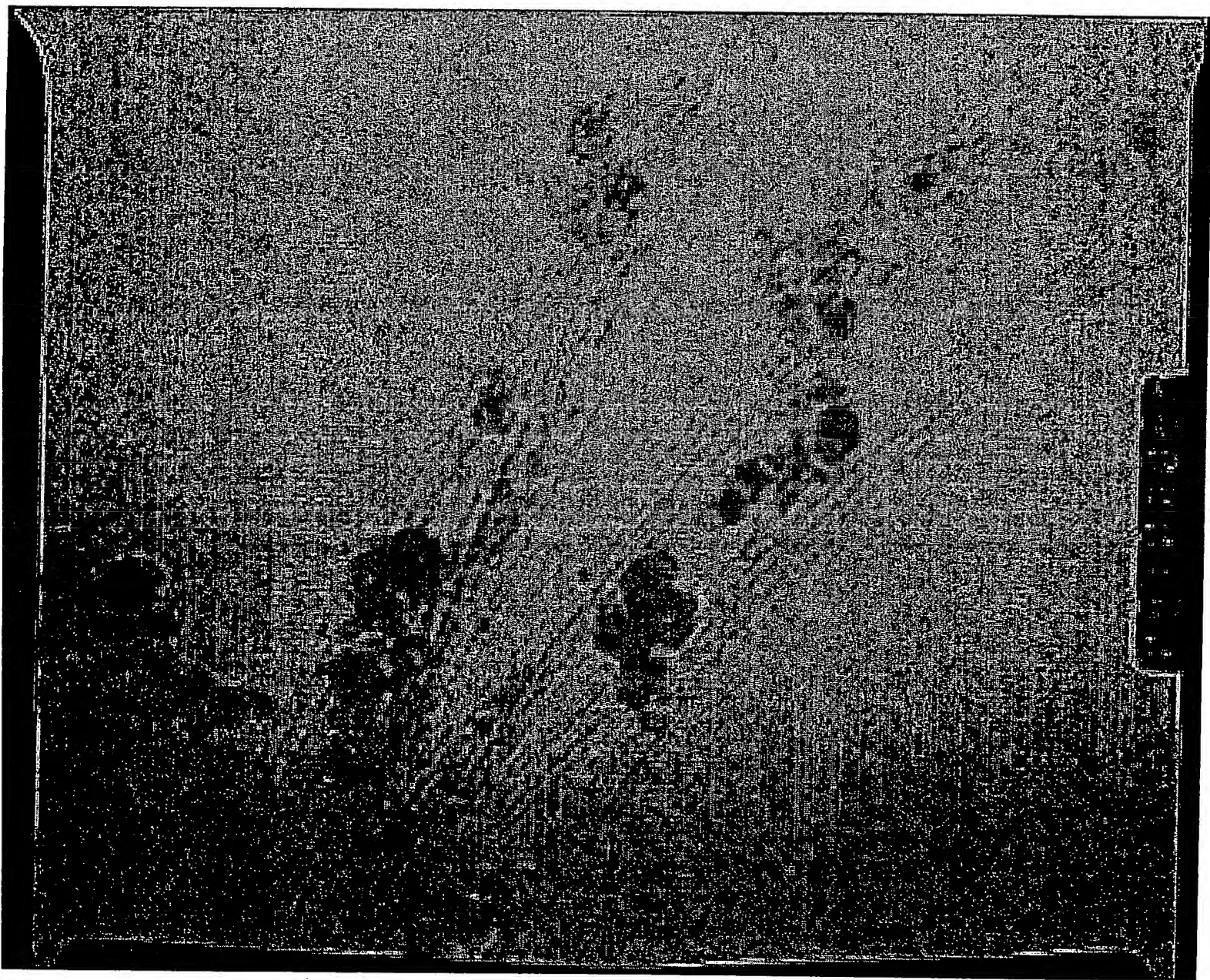
Left: crispy powder, burned
wellsSEM pWS: Curly bundles $\phi \sim 100nm$ embedded
in amorph, very long ($> 5-10\mu m$)TEM: Thin bundles of 5-20 tubes & some
separate thick tubes of $\sim 4-5\mu m \phi$
Most of tubes have 2 walls!Some are SWNTs (thick $\phi \sim 4-5\mu m$)(The system selectively produces
double-walled nanotubes in abundance!)

A. Moradshahi

Read & understood

Lowe

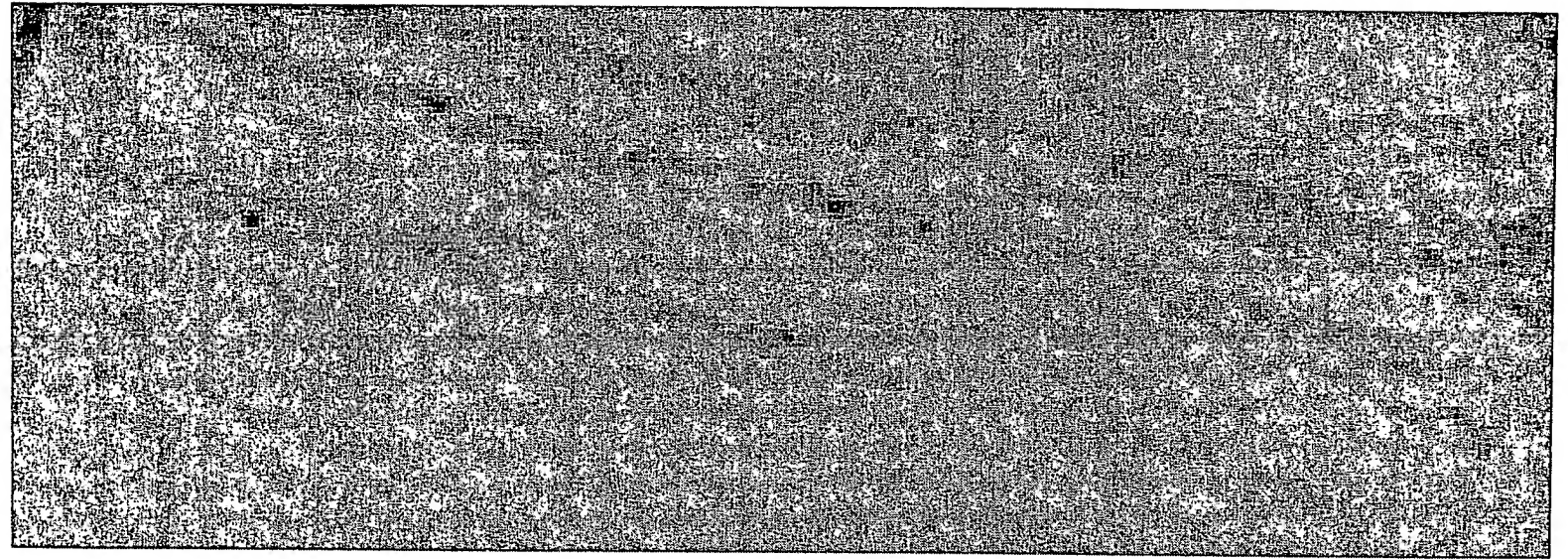
TEM image of the product of Run CF39



TEM image of the product of Run CF39



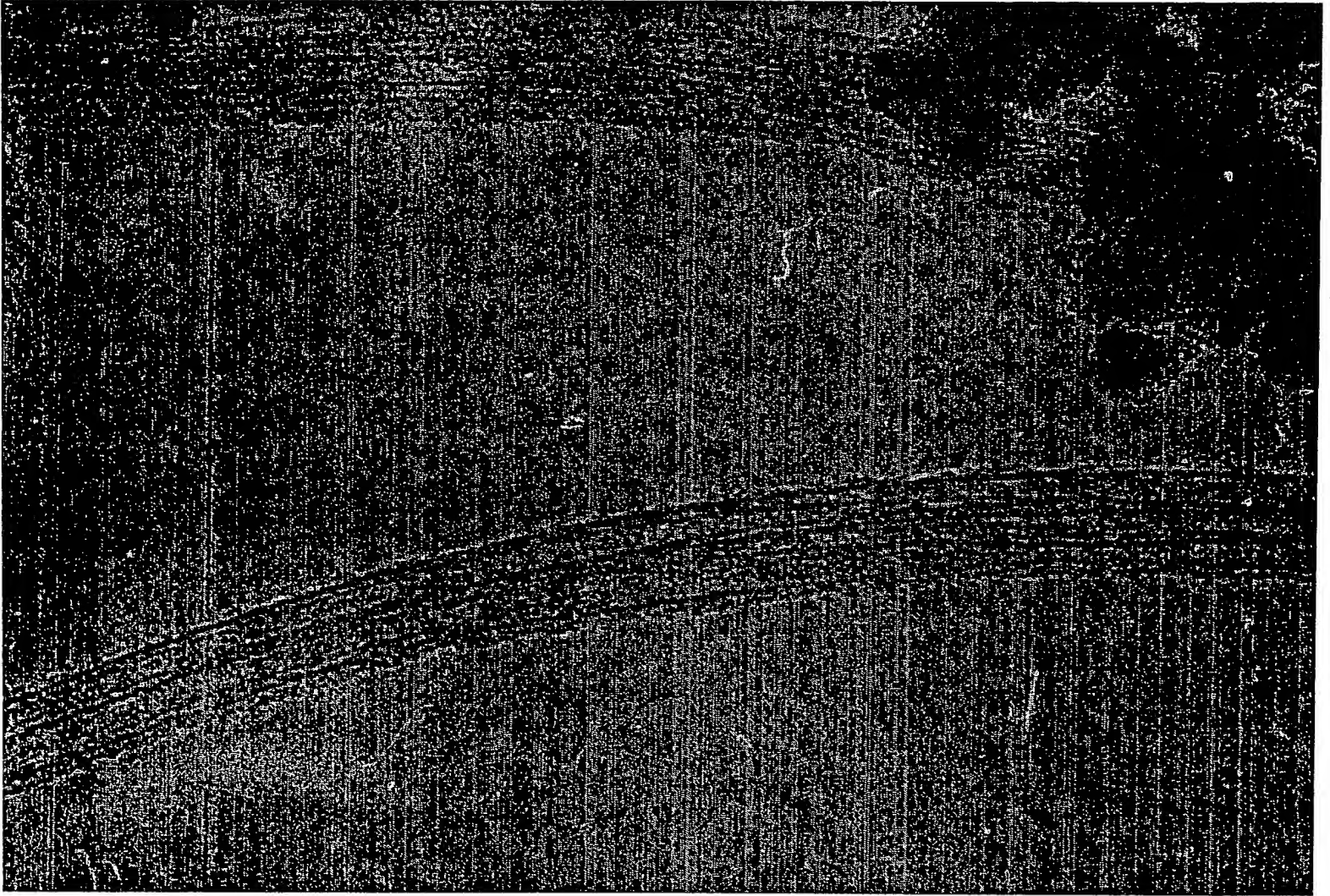
TEM image of the product of Run CF39



TEM image of the product of Run CF39



TEM image of the product of Run CF39



TEM image of the product of Run CF39



Message for Dr.A.P.Moravsky

Subject: Message for Dr.A.P.Moravsky

Date:

From: "Elena P. Krinichnaja" <elkrina@icp.ac.ru>

Organization: IPCP

To: "moravsky@Opus1.com" <moravsky@Opus1.COM>

Саша, здравствуй!

ТЭМ (кобальт, никель, железо и сульфид железа) двухслойные нанотрубки, диаметр 3.5-5.2 нм, расстояние между стенками 0.42+/-0.02 нм. ←
Внутренние каналы нанотрубок частично заполнены, по всей видимости, аморфным углеродом. Помимо нанотрубок и каталитических частиц в образце присутствуют различные формы углерода, структура которых на данный момент времени не анализировалась.

Елка..

П.С. Ты когда-нибудь ответишь мне?

E-mail message

for Dr. A.P.Moravsky from Dr.E.P.Krinichnaja

Translation into English

Hello, Alex!

TEM (cobalt, nickel, iron and iron sulfide catalyst) has shown: double-walled nanotubes of 3.5 – 5.2 nm diameters, with the distance between the walls 0.42 ± 0.02 nm.

Internal channels of nanotubes are partially filled, most likely, with amorphous carbon.

Besides the nanotubes and catalytic particles, there are present various forms of carbon with the structure that was not yet analyzed.

Elena.

P.S. Waiting for your reply.



Double-walled carbon nanotubes fabricated by a hydrogen arc discharge method

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Abstract

Double walled carbon nanotubes (DWNTs) were obtained by the arc discharge technique in an atmosphere of Ar and H₂ mixture (1:1/v:v) at 350 Torr. The catalyst was prepared from a mixture of Ni, Co, Fe and S powders heated in an inert gas atmosphere at 500°C for 1 h. High resolution electron microscopy (HREM) revealed that the dominant type of obtained nanotubes were DWNTs with outer diameter in the range of 1.9–5 nm and inner tube diameters in the range 1.1–4.2 nm. As a rule, the DWNT tubes form into bundles. Occasionally single walled nanotubes (SWNTs) were observed by HREM although Raman spectroscopy did not reveal the presence of significant quantities of these tubules in the bulk product.
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Keywords: A. Carbon nanotubes; B. Plasma deposition; C. Transmission electron microscopy; Raman spectroscopy; D. Microstructure

1. Introduction

Selective production of specific types of carbon nanotubes (NTs) is one of the principal goals of NT synthesis technology. This is necessary for establishing the correlation between different types of physical properties (electro- and heat conductivity, mechanical rigidity and so on) and the obtained NT type, for testing the validity of the theoretical predictions regarding their respective properties and also for the achievement of the specified properties within bulk purified samples.

The synthesis of single-walled NTs (SWNTs) in an atmosphere of hydrogen by arc discharge techniques was recently published [1,2]. As a catalyst, a mixture of Ni, Co,

Fe and FeS was evaporated together with the carbon feedstock. The reactor construction was modified relative to the conventional arc discharge method normally used for SWNTs production [3–5]. Instead of a catalyst-filled anode, a set of round grooves on the anode surface was filled with a mixture of carbon and the catalyst powder. The cathode axis was also oriented at an angle to the anode axis instead of the 'end to end' anode–cathode geometry normally employed. In these conditions, high quality SWNTs were obtained. The specimens were investigated by resonant Raman scattering measurement and by HREM. Three well-resolved peaks at 1593 cm⁻¹, 1576 cm⁻¹ and 1569 cm⁻¹, can be assigned to a splitting of the E_{2g} mode of graphite considered should be characteristic of SWNTs [6]. HREM observations revealed that, for the SWNT bundles, the mean SWNT diameter was 1.85 nm [1].

We performed experiments in conditions very close to those in Ref. [1], except that the normal configuration of

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electrode construction and orientation (i.e. 'end to end') was used. Under these conditions, as we describe here, DWNTs were formed as the dominate component in the product mixture.

2. Experimental

2.1. Preparation of nanotubes

To produce NTs, arc discharge evaporation of a metal-graphite electrode was used. The anode was a graphite rod (8.2 mm in diameter) with a drilled channel (3.2 mm in diameter and 140 mm in length) filled with catalyst. The cathode was a graphite rod 10 mm in diameter and 25 mm in length. The catalyst was prepared in the following way: a mixture of Ni, Co and Fe powder metals (particle sizes 2–5 μm) and elemental S was ground in a mortar and then heated in an inert gas atmosphere at 500°C for 1 h. The conglomerate was then ground in a ball mill to a micron particle size and then intimately mixed with carbon powder. A 3.2-mm channel drilled in the graphite anode was tightly filled with this mixture. The content of elements in the composite anode with respect to C was: Ni 2.6, Co 0.7, Fe 1.45 and S 0.75 at.% and so matched the metal and sulfur content of the anode materials used for the synthesis described in Ref. [1].

The process of catalyst synthesis was carried out with an arc current of 75–80 A in an atmosphere of Ar and H_2 mixture (1:1/v:v) at 350 Torr with a 410-Torr partial vacuum. The voltage drop across the gap was 26–28 V and the gap width kept constant and equal to 2 mm. The duration of the arc evaporation process was about 40 min. Two types of carbon material were obtained: (1) the product deposited on the reactor walls; and (2) the remaining product deposited on electrodes and cathode deposit as an elastic web-like product.

2.2. Electron microscopy

Specimens for TEM and HREM were prepared by dispersing the web-like products collected from the chamber wall and between the electrodes in acetone in an ultrasonic bath. A drop of the suspension was put on a lacy carbon film supported by a copper grid. Specimens were investigated in a Philips EM-430ST electron microscope operated at 200 kV, in a JEOL JEM-4000EX operated at 400 kV and JEM-3000F instrument operated at 300 kV with Oxford Instrument ISIS 300™ EDX system and Gatan (1 k \times 1 k) CCD camera. To calibrate the magnification in the EM-430ST, lattice fringes obtained from Si crystals in [110] orientation were used as a reference standard.

2.3. Raman spectroscopy

Raman measurements were performed with a Jobin

Yvon S-3000 spectrometer equipped with a triple monochromator and operated in microRaman configuration. Ar^+ -laser irradiation with a wavelength of 514.5 nm was used for spectra excitation. Since the material was very sensitive to heating, the measurements were carried out at low laser power (i.e. less than 20 mW).

3. Results

A general view of the specimen is shown in Fig. 1a. The nanotubes form readily into bundles with discrete tubules clearly visible on the periphery. The observed length of these tubes in bundles is more than 1 μm . The HRTEM micrographs (Fig. 1b) revealed that most of the tubules have a specific structure and consisted of two concentric cylindrical graphene layers, that is they are double wall nanotubes (DWNTs). In general, the DWNTs were measured with outer diameters of between 1.9 and 5 nm while the diameter of the inner tube varied between 1.1 and 4.2 nm. Along with the bundles, discrete separated DWNTs were also observed. These discrete DWNTs are easily distorted (Fig. 2). That means they are mechanically less rigid in comparison with single wall nanotubes (SWNTs), probably because of their larger diameter. The tubule diameter in Fig. 3a varied between 3 nm and 4.95 nm. For the DWNT in Fig. 3b, a variation of the tubule diameter from 3.75 to 5.0 nm along the length of the tube is visible at intervals of between 30 and 35 nm. Some DWNTs have rounded double layered terminating caps at the end (Fig. 3c). Notably, the interlayer distance measured in the tubule wall images taken at 200 kV and 400 kV is 0.39 ± 0.02 nm which is larger than that usually observed for multilayered NTs (0.34 nm). A micrograph of a DWNT obtained in the 3000F microscope and, from the indicated wall region, contrast density profile are shown in Fig. 6. The latter reveals an interlayer separation of 0.403 nm. A range of similar measurements obtained from other DWNTs revealed that this wall spacing was typical.

Often catalytic particles (CPs) surrounded by one to several graphene layers were observed (see Fig. 1b, Fig. 2 and Fig. 4) with interlayer distances of the order of 0.36 ± 0.02 nm. HRTEM images of CP vary in lattice images. There is indirect evidence that during the formation of the DWNTs, the CPs are in the melt state since some DWNTs are filled with dense material (Fig. 3d). EDX spectrum from CP revealed peaks corresponding to Co, Ni and Fe. Traces of S were also revealed (see, for example Fig. 5).

In some DWNTs, fragments of third and fourth (Fig. 3a) layers were observed. At the same time, SWNTs were seldom observed (Fig. 4). Their diameters were in general found to be close to the inner tube diameter in the observed DWNTs (i.e. 2.7–4.7 nm). It is believed that such SWNTs could be considered as DWNTs in which the formation of the second layer does not occur or was

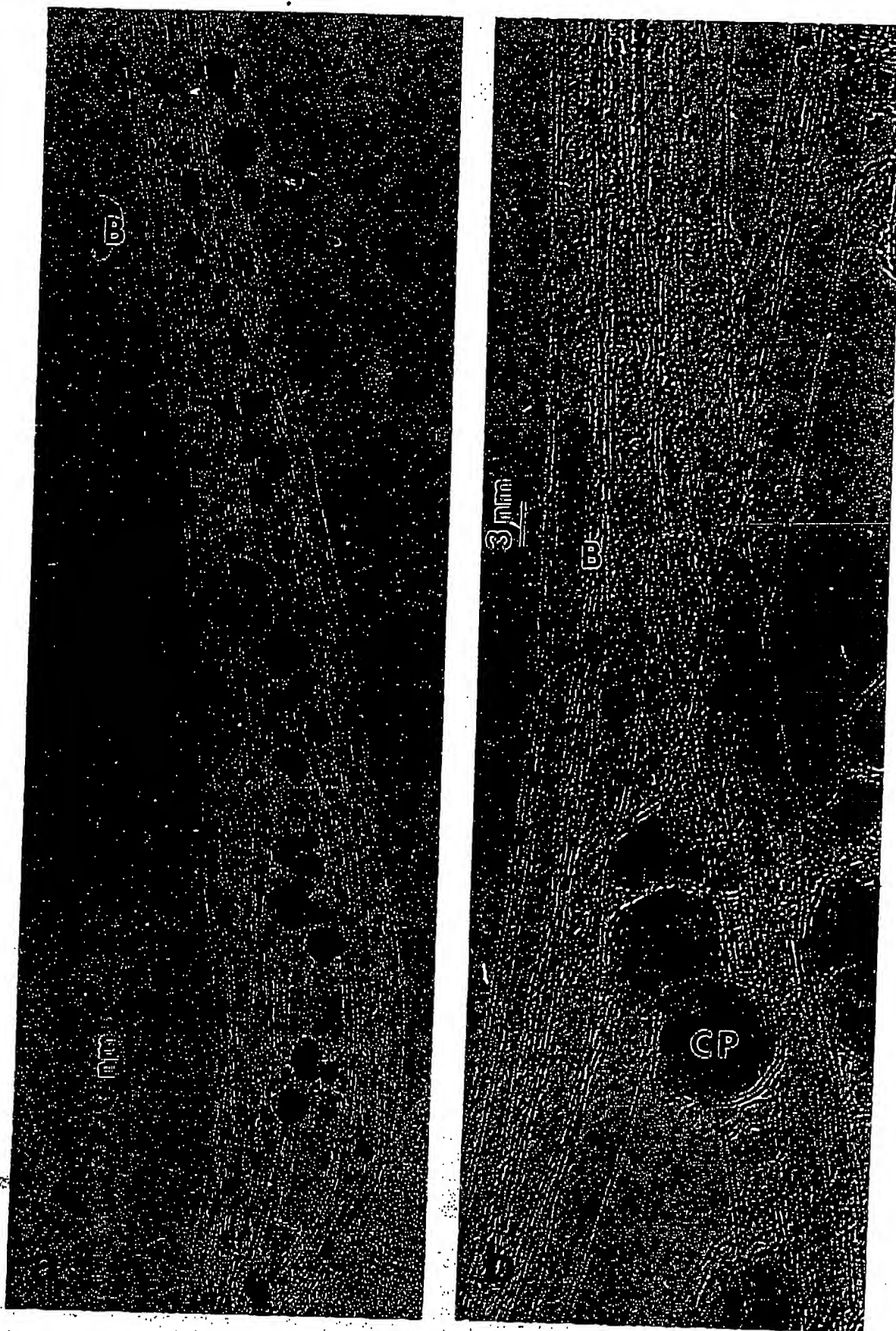


Fig. 1. (a, b) General views (400 kV) showing bundles (B) of straight double walled nanotubes (DWNTs); CP, catalytic particle.

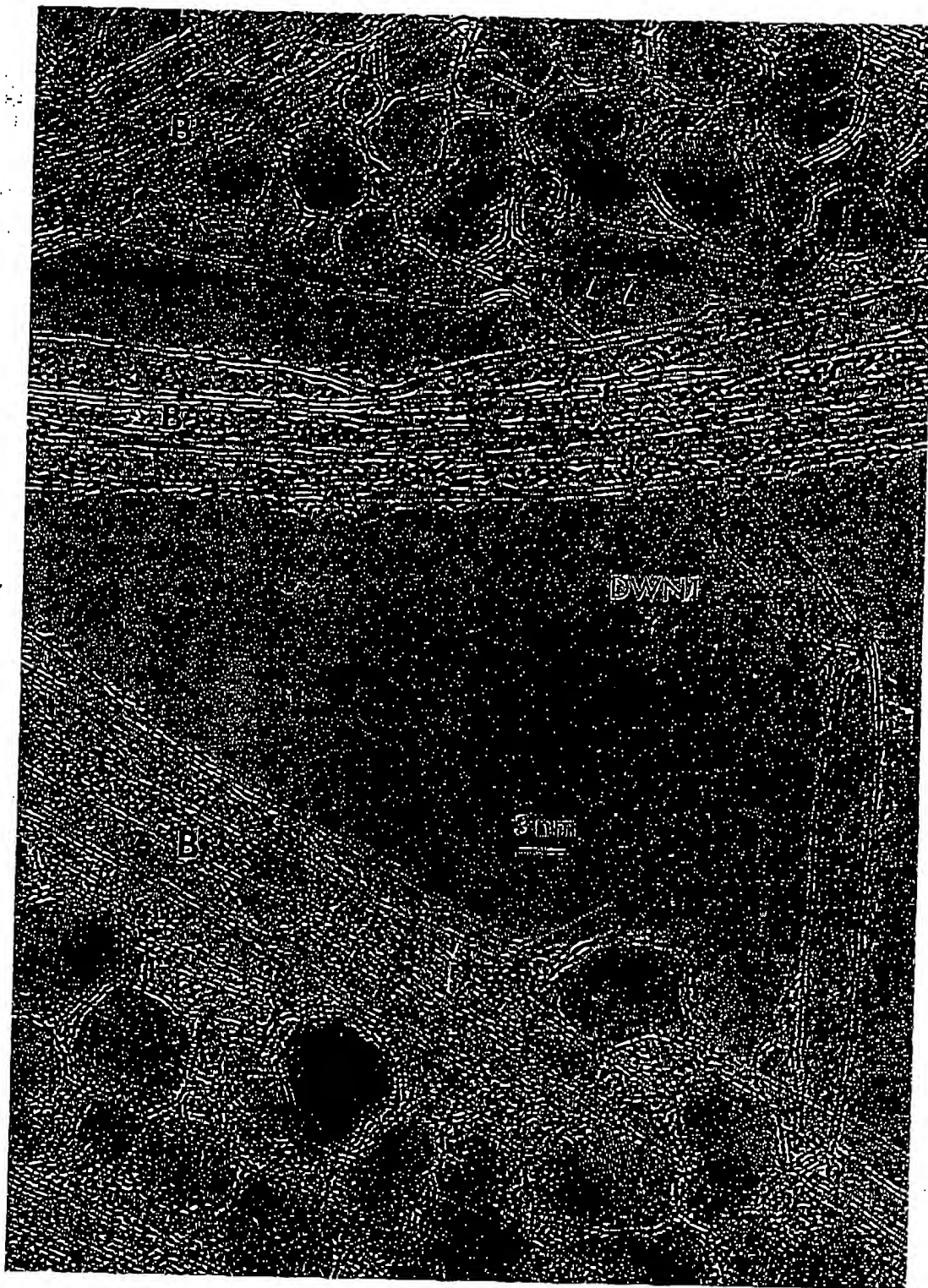


Fig. 2. General view (400 kV) showing bundles (B) and separate DWNTs. The regions of DWNT distortion are marked by large arrows. Small local variations of nanotube diameter are marked by small arrows.

incomplete. A few NTs were observed that conformed to small diameter (i.e. 1.7–2 nm) SWNTs.

Along with DWNTs diameter, variations were caused by tube deformations resulting, in some cases, in local small variations (10–15%) in diameter. These variations are

sometimes observed only for inner tubes (Fig. 2 and Fig. 4). They are visible in SWNT images as well (Fig. 6). In our opinion local variations are connected with the process of nanotube growth.

The Raman spectrum (Fig. 7) was dominated by the

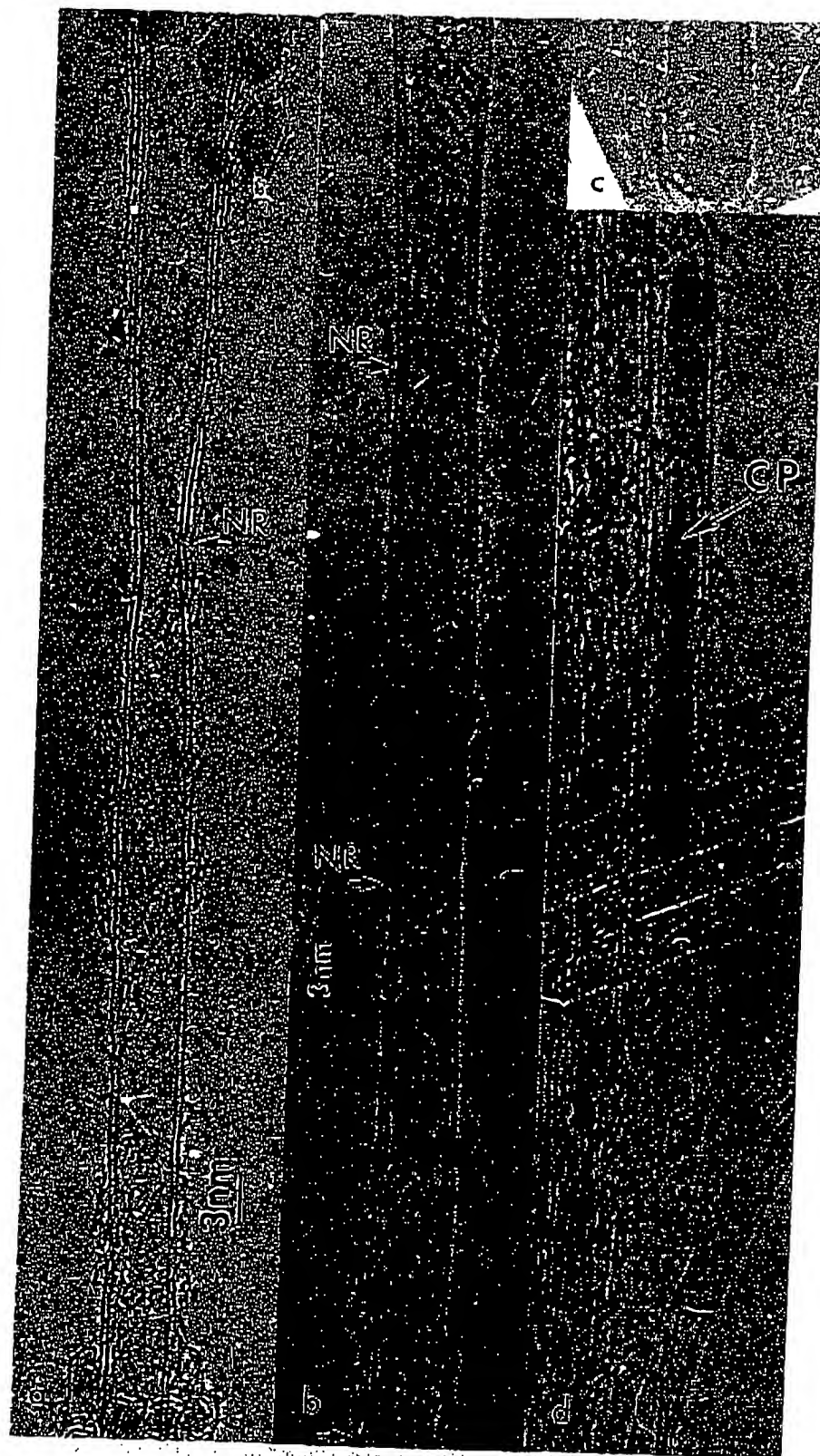


Fig. 3. Separate nanotubes of different types: (a) DWNT with varying diameter (400 kV). Narrow region (NR) is arrowed. Small fragments of the third and fourth layers are visible (marked by large arrows). (b) DWNT with pseudoperiodically varying diameter (200 kV). Several narrow regions (NR) are arrowed. Some forms of amorphous carbon material are observed both inside and outside the tube. (c) DWNT with rounded cpd cap (200 kV). (d) DWNT filled with dense material, probably CP (200 kV).

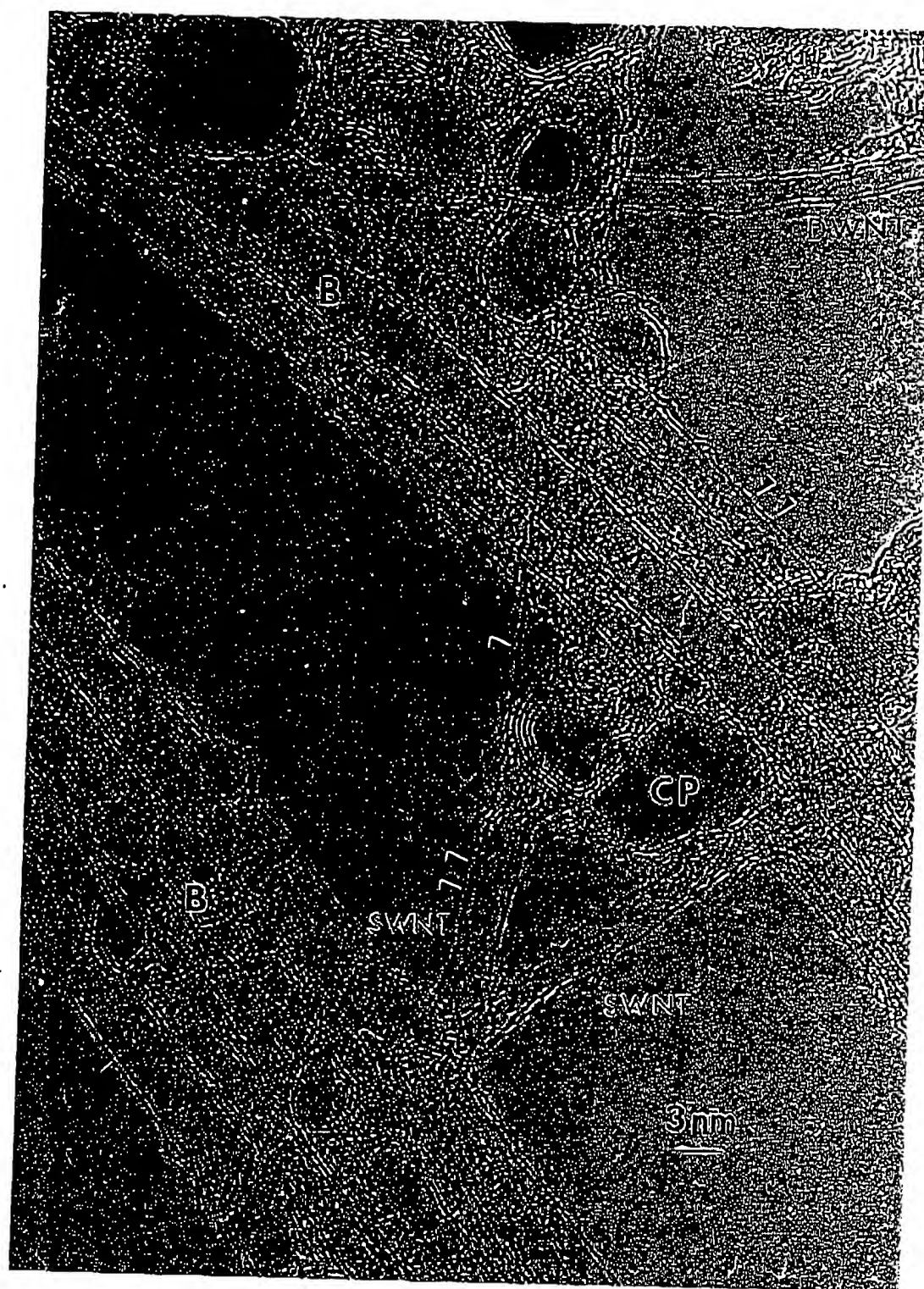


Fig. 4. General view of DWNTs (400 kV) showing bundles (B), separate DWNT with relatively small diameter (2.2–2.5 nm) and SWNTs. Small local variations in the DWNT and SWNT diameters are marked by small arrows.

band at 1582 cm^{-1} with a full width at half maximum (FWHM) of 50 cm^{-1} . This feature may be assigned both to well-crystallized graphite [7] and to multiwall carbon nanotubes [8]. Unfortunately there are no characteristic 'fingerprint' features for multiwall carbon nanotubes in the

Raman spectrum which enables them to be distinguished from graphitic carbon in the Raman spectrum. This is the case only for single-wall carbon nanotubes which demonstrate a splitting of the tangential mode (i.e. near 1592 cm^{-1}) and an appearance of the 'breathing' mode with a

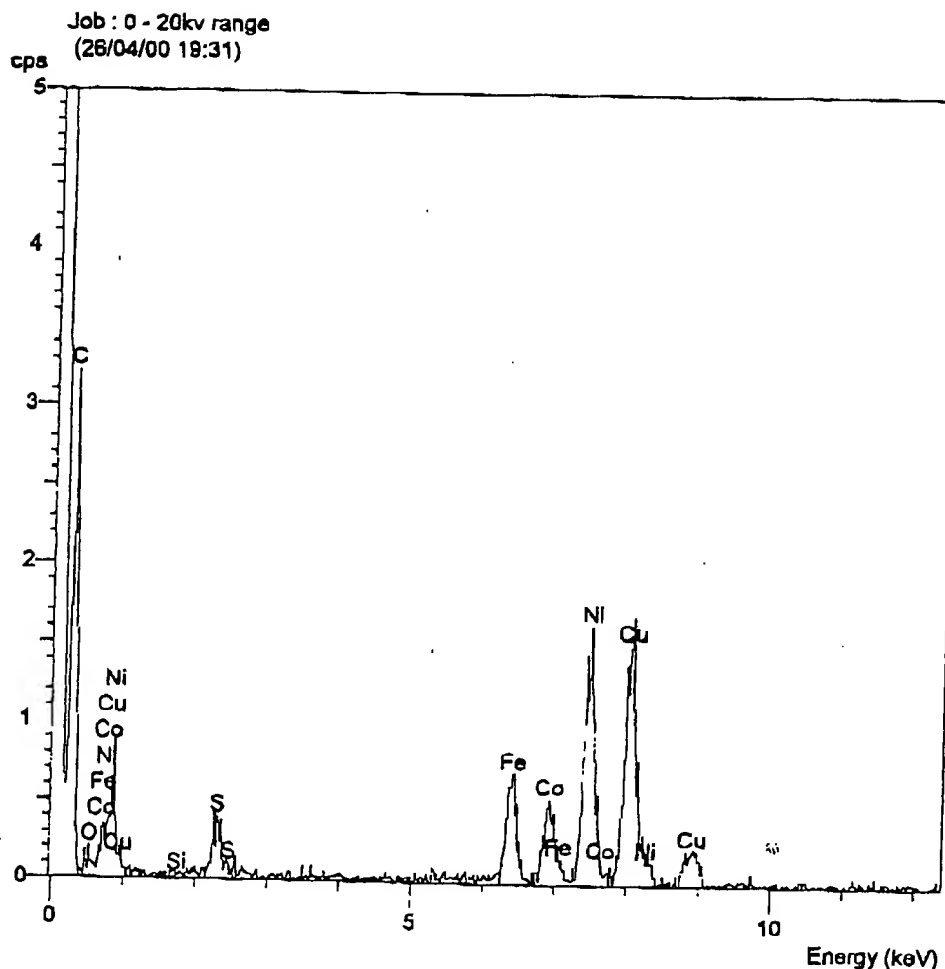


Fig. 5. EDX spectrum.

frequency strongly depending on the tube diameter in the range $100\text{--}200\text{ cm}^{-1}$ [9,10]. These features are observed for nanotubes with a diameter less than 2 nm [11]. For larger nanotubes they disappear due to overlapping of maxima in the nanotube electronic density.

4. Discussion

The dominant type of structure found in our product were DWNTs. Within the observed tubules, there was a clear distribution of diameters suggesting that a number of different (n,m) conformation nanotubes were present in the inner and outer tubules making up the composite DWNT tubules. A few nanotubes were observed with third and fourth layers but these were usually incomplete or damaged. This phenomenon could possibly be accounted for by the employed experimental conditions (for example the presence of hydrogen).

As mentioned above, a small number of SWNTs were observed in the specimen and their minimum diameter was found to be ca. 1.7 nm. The small quantity of these nanotubes observed within the specimen also accounts for

the absence of the corresponding peaks within the Raman spectrum. Peaks corresponding to SWNTs were reported in Ref. [1] in which a far higher proportion of SWNTs was observed in the product. It might have been expected that for the DWNTs the signals from both shells would have been seen in the Raman spectrum. However in the case of our product the average tube diameter was too large to observe this effect.

It has been shown that addition of sulfur to the starting mixture of C and Co significantly increased the yield of SWNTs found in the arc discharge product [12]. In addition a greater spread in the size distribution of the observed SWNTs was reported and, occasionally, single DWNTs were observed. It was suggested that sulfur may affect the kinetics by removing terminating species from the growing ends of the tubes. Another possibility is that sulfur species could bind to and stabilize the ends of large diameter tubes. In the modified experiment described here the catalytic arc-discharge system has selectively produced DWNTs as the main product collected from electrodes in the reactor (although not from the cooled walls).

It is necessary to mention that even the smallest deviation from the synthesis conditions described above

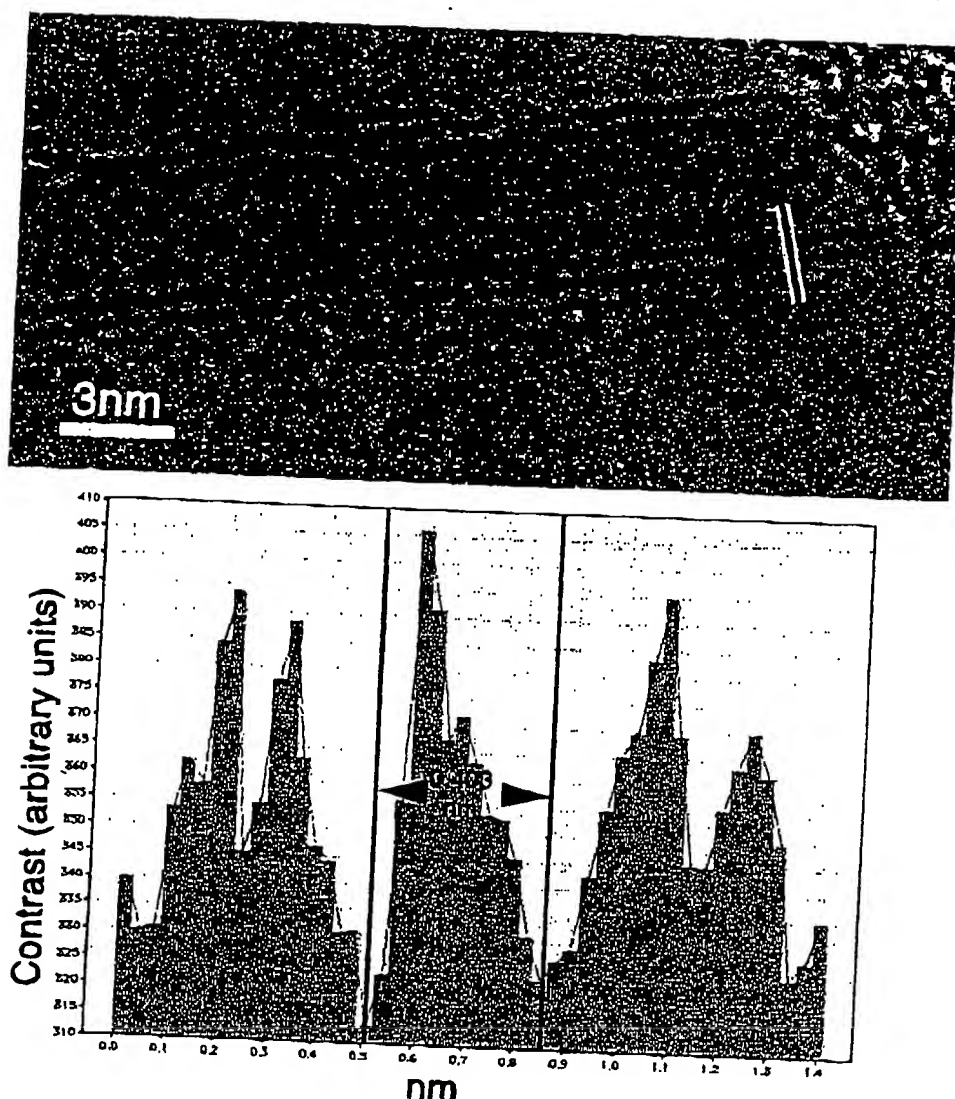


Fig. 6. DWNT micrograph (top) obtained in a JEM-3000F microscope 300 kV and a contrast density profile (bottom) of the nanotube wall, revealing an interlayer distance 0.403 nm.

led to a decrease in the formation of DWNTs and resulted in a domination of SWNTs in the product mixture. In general the amount of DWNTs in the product varied from ~10–20 wt.% to ~50–70 wt.%. It appears therefore that the conditions and arcing geometry of the experiment are close to optimal for the selective and effective production of DWNTs via the arc discharge method.

To optimize the process of SWNTs synthesis, a series of experiments in an arc-discharge reactor was performed with a wide range of Co:Ni:Fe catalyst compositions with controlled arc-discharge parameters [13]. In all cases SWNTs were produced in the synthesis product and only a very few DWNTs were observed. This begs the question: what has drastically changed the morphology of the condensation products in the conditions used in the present paper? To clarify this we performed additional experiments that showed that sulfur addition to the metallic catalyst when the arc-discharge process was carried out only

increased the SWNTs yield, but did not lead to the formation of DWNTs. This finding is in good agreement with published results on the influence of sulfur on the catalytic synthesis of carbon nanotubes. It was shown previously [12] that sulfur addition to carbon and cobalt mixture led to an increase in the yield of SWNTs. In our case, sulfur addition resulted in an increase in the nanotube diameter distribution (1–6 nm).

We have also performed experiments to reveal the role of hydrogen. It was shown that in the products formed from arc discharge in H_2 /Ar atmosphere DWNTs appeared and even predominated. This indicates that the presence of sulfur in the catalyst is not critical for the formation of DWNTs. In order to understand DWNTs appearance when He atmosphere is replaced by a mixture of Ar and H_2 , it is necessary to explain how the process is changed.

It is known that at similar arc parameters (i.e. current and gas pressure) the carbon plasma temperature in Ar is

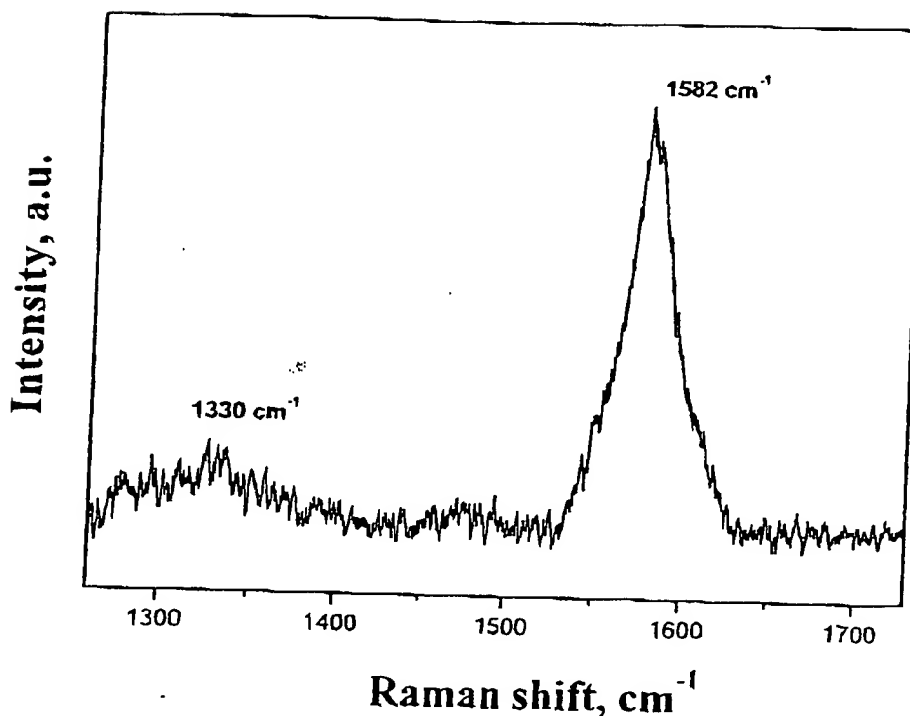


Fig. 7. Raman spectrum of the DWNTs product.

essentially lower than in the He atmosphere. The anode carbon in Ar is evaporated only slowly during arcing. Apparently, this is due to the larger value of the He ionization energy (24.5 eV) compared to the Ar one (15.76 eV). This means that a higher plasma temperature is required in case of He in order to produce the same equilibrium concentration of current carriers. If the helium arc temperature is 3600–3900 K then this is sufficiently large for carbon evaporation [14]. The Ar arc temperature in the same conditions can be approximated to 2400–2600 K which is indeed not hot enough for anode graphite evaporation [15]. However if H_2 is added to the Ar atmosphere, the anode evaporation is very rapid. The gas temperature in the arc in this case does not change relative to that obtained in pure argon. This means that the anode graphite, when H_2 is added, is evaporated in the form of hydrocarbon fragments, radicals and molecules. Therefore, under these conditions, the arc-discharge process is transformed into a gas-phase analog of a catalytic pyrolysis process with a 'floating' catalyst.

The complete pattern of the process of carbon formation in the arc-discharge reactor when H_2 is added is obviously more complicated in comparison with that which occurs in an inert atmosphere. At present, not all of the details are clear, but some phenomena are interpretable. For example, from the available thermodynamic data it is possible to estimate that, under the present conditions of evaporation (2400–2600 K), the equilibrium concentrations of polyene radicals of the form $C_{2n}H^{\cdot}$, with $n=1, 2, \dots$ are only a small fraction of the overall polyene concentrations itself. The reason for this is the large energy of the C–H bond in

the polyene molecule, which is ~ 120 kcal/mol. Therefore, in an arc gas and in an Ar and H_2 atmosphere at 2400–2600 K the large proportion of carbon clusters is in the form of acetylene and polyene molecules of the form $C_{2n}H_2$.

If one temporarily puts aside the role of the catalyst and the influence of the arc discharge atmosphere then, structurally, the different types of NTs can be aligned in the following series in accordance with synthesis temperature: SWNTs > MWNTs > CLNTs > NFs (NFs = nanofilaments). According to synthesis temperature DWNTs must be located somewhere between MWNTs and SWNTs. It is noticeable that the thermal conductivity of NTs is decreased passing from SWNTs to CLNTs and NFs.

Acknowledgements

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References

- [1] Liu C, Cong HT, Li F, Tan PH, Cheng HM, Lu R et al. Semi-continuous synthesis of single-walled carbon nanotubes by a hydrogen arc discharge method. *Carbon* 1999;37:1865–8.
- [2] Liu C, Fan YY, Liu M, Cong HT, Cheng HM, Dresselhaus

- MS. Hydrogen storage in single-walled carbon nanotubes at room temperature. *Science* 1999;286:1127–9.
- [3] Iijima S, Ichihashi T. Single-shell carbon nanotubes of 1-nm diameter. *Nature* 1993;363(6430):603–5.
- [4] Bethune DS, Kiang CH, de Vries MS, Gorman G, Savoy R, Vazquez J et al. Cobalt-catalysed growth of carbon nanotubes with single-atomic-layer-walls. *Nature* 1993;363(6430):605–7.
- [5] Thess A, Lee R, Nikolaev P, Dai HJ, Petit P, Robert J et al. Crystalline ropes of metallic carbon nanotubes. *Science* 1996;273(5274):483–7.
- [6] Kasuya A, Sasaki Y, Saito Y, Tohji K, Nishina N. Evidence for size-dependent discrete dispersions in single-wall nanotubes. *Phys Rev Lett* 1997;78(23):4434–7.
- [7] Tuinstra F, Koenig JL. Raman spectrum of graphite. *J Chem Phys* 1970;53:1126–30.
- [8] Zhao X, Ando Y. Raman spectra and X-ray diffraction patterns of carbon nanotubes prepared by hydrogen arc discharge. *Jpn J Appl Phys* 1998;37(part 1, N9A):4846–9.
- [9] Rao AM, Richter E, Bandow S, Chase B, Eklund PC, Williams KA et al. Diameter-selective Raman scattering from vibrational modes in carbon nanotubes. *Science* 1997;275:187–91.
- [10] Obratsova ED, Yurov VYu, Shevlyuga VM, Baranovsky RE, Nalimova VA, Kuznetsov VL et al. Structural investigations of close-packed single-wall carbon nanotube material. *Nanostruct Mater* 1999;11(3):295–306.
- [11] Fang SL, Rao AM, Eklund PC, Nikolaev P, Rinzler AG, Smalley RE. Raman scattering study of coalesced single walled carbon nanotubes. *J Mater Res* 1998;13(9):2405–11.
- [12] Kiang C-H, Goddard III WA, Beyer R, Salem JR, Bethune DS. Catalytic synthesis of single-layer carbon nanotubes with a wide range of diameters. *J Phys Chem* 1994;98(26):6612–8.
- [13] Loutfy RO, Lowe TP, Hutchison JL, Kiselev NA, Zakharov DN, Krinichnaya EP et al. A 'dissolution-precipitation' model for the catalyst of SWNT growth. In: Abstracts of Invited Lectures & Contributed Papers, 4th Biennial International Workshop in Russia 'Fullerenes and Atomic Clusters' (IWFAC'99), St. Petersburg, 1999.
- [14] Krestinin AV, Moravsky AP. Mechanism of fullerene synthesis in the arc reactor. *Chem Phys Lett* 1998;286:479–84.
- [15] Abrahamson J. Graphite sublimation temperatures, carbon arcs, and crystallite erosion. *Carbon* 1974;12:111–41.